Yttrium alkyl and benzyl complexes with amino-amidinate monoanionic ancillary ligands

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Published in:
Organometallics

DOI:
10.1021/om0001063

Publication date:
2000

ORGANOMETALLICS OM000 1063

Supporting material to:

"Yttrium Alkyl and Benzyl Complexes with Amino-Amidinate Monoanionic Ancillary Ligands"
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Part I: Crystal structure determination of [*t*-BuC(N*i*-Pr)$_2$]Y[CH(SiMe$_3$)$_2$]$_2$ClLi(THF)$_3$ (1).

Abstract. $C_{37}H_{85}ClLiN_2O_3Si_4$, M = 849.74, monoclinic, $P2_1/n$, a = 13.422(1), b = 17.781(1), c = 21.269(1) Å, β = 91.62(1)°, V = 5074.0(5) Å^3, Z = 4, D_x = 1.112 g cm^{-3}, λ(MoK$\bar{\alpha}$) = 0.71073 Å, μ = 13.3 cm^{-1}, F(000) = 1840, T = 130 K, $wR(F^2)$ = 0.2060 for 9386 reflections with $F_o^2 \geq 0$ and 461 parameters, 66 restraints and $R(F)$ = 0.0793 for 4912 reflections obeying $F_o \geq 4.0\ \sigma(F_o)$ criterion of observability. The asymmetric unit consists of one molecule of the title compound.

Experimental

X-ray diffraction: Crystal and Molecular Structure.

A plate-shaped colorless crystal, of approximate size 0.10 x 0.25 x 0.50 mm., used for characterization and data collection was glued on top of a glass fiber by using inert-atmosphere handling techniques and was transferred into the cold nitrogen cold stream of the low temperature unit[1] mounted on an Enraf-Nonius *CAD-4F*[2] diffractometer, interfaced to a *INDY* (*Silicon Graphics*) *UNIX* computer (Mo tube, 50 kV, 40 mA, monochromated Mo-K$\bar{\alpha}$ radiation, $\Delta\omega = 0.90 + 0.34$ tg θ).

Unit cell parameters[3] and orientation matrix were determined from a least-squares treatment of the *SET4*[4] setting angles of 21 reflections in the range 15.00° < θ < 20.33°. The unit cell was identified as monoclinic; reduced cell calculations did not indicate any higher metric lattice symmetry.[5] The space group $P2_1/n$ was derived from the systematic extinctions. Examination of the final atomic coordinates of the structure did not yield extra metric symmetry elements.[6,7]

The intensities of three standard reflections, monitored every three hours of X-ray exposure time, showed no greater fluctuations during data collection than those expected from Poisson statistics. 360° ψ-scans for reflections close to axial showed variation in intensity of less than 15% about the mean value. Intensity data were corrected for Lorentz and polarization effects, scale variation, but not for absorption and reduced to F_o^2.[8]

The structure was solved by Patterson methods and extension of the model was accomplished by direct methods applied to difference structure factors using the program *DIRDIF*.[9] The positional and anisotropic thermal displacement parameters for the non-hydrogen atoms were refined. Refinement was complicated by a disorder problem: from the solution it was clear that the three THF molecules coordinated to Li were highly disordered. Some carbon atoms of the THF ligands showed unrealistic thermal displacement parameters suggesting some degree of disorder, which is in line with the

refinement, which did not give much improvement. So ultimately similarity restraints were used for the refinement of the THF ligands (the disorder is compensated by some huge thermal displacement parameters).

The hydrogen atoms were included in the final refinement riding on their carrier atoms with their positions calculated by using sp^3 hybridization at the C-atom as appropriate with $U_{iso} = c \times U_{equiv}$ of their parent atom, where c = 1.2 for the non-methyl hydrogen atoms and c = 1.5 for the methyl hydrogen atoms and where values U_{equiv} are related to the atoms to which the H atoms are bonded. The methyl-groups were refined as rigid groups, which were allowed to rotate free.

Final refinement on F^2 carried out by full-matrix least-squares techniques converged at $wR(F^2)$ = 0.2060 for 9380 reflections with $F_o^2 \geq 0$ and $R(F)$ = 0.0793 for 4912 reflections with $F_o \geq 4.0\ \sigma(F_o)$ and 461 parameters and 66 restraints.

The final difference Fourier map was essentially featureless: no significant peaks (0.99(10) e/Å^3) having chemical meaning above the general background were observed.

The positional and anisotropic thermal displacement parameters for the non-hydrogen atoms were refined on F^2 with full-matrix least-squares procedures minimizing the function $Q = \Sigma_h\ [w(|F_o^2 - kF_c^2|)^2]$, where $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$, $P = [\max(F_o^2,0) + 2F_c^2] / 3$, F_o and F_c are the observed and calculated structure factor amplitudes, respectively; a and b were refined. Reflections were stated observed if satisfying $F^2 > 0$ criterion of observability.

Neutral atom scattering factors and anomalous dispersion corrections were taken from *International Tables of Crystallography*.[10] All calculations performed on the HP9000/735 computer at the University of Groningen with the program packages *SHELXL*[11] (least-square refinements), *PLATON*[12] (calculation of geometric data and the *ORTEP* illustrations) and a locally modified version of the program *PLUTO*[13] (preparation of illustrations).

The monoclinic unit cell contains four discrete units of each type of the title compound separated by normal van der Waals distances.[16]

No missed symmetry (*MISSYM*) were detected by procedures implemented in *PLATON*.[17]

The unit cell contains a potential solvent area (voids) of 104.7 Å^3.[18]

Table 1.

a. Crystal data and details of the structure determination.

Moiety_ Formula	$C_{37}H_{85}ClLiN_2O_3Si_4$
Formula_Weight, g.mol^{-1}	849.74
Crystal system	monoclinic
Space group, no.[19]	$P2_1/n$, 14
a, Å	13.422(1)
b, Å	17.781(1)
c, Å	21.269(1)
β, deg	91.62(1)
V, Å^3	5074.0(5)
Formula_*Z*	4
SpaceGroup_*Z*	4
Z' (= Formula_Z / SpaceGroup-*Z*)	1
ρ_{calc}, g.cm^{-3}	1.112
F(000), electrons	1840
μ(Mo K), cm^{-1}	13.3
color, habit	colorless, plate
Approx. crystal dimension, mm	0.10 x 0.25 x 0.50

b. Data collection.

Radiation	Mo K
Wavelength, Å	0.71073
Monochromator	Graphite
Temperature, K	130
θ range; min. max., deg	1.15, 25.0
ω/2θ scan, deg	$\Delta\omega = 0.90 + 0.34$ tg θ
Index ranges	h: -16→16; k:0→21; l: 0→25
Crystal-to-receiving-aperture-distance, mm	173
Horizontal-, vertical-aperture, mm	3.2 + tg θ; 4.0
Reference reflections,	024, 2.1
r.m.s. dev. in %	-1-33, 0.23
	3-12, 0.61
Drift correction	1.000 - 1.037
X-ray exposure time, h	132.0
Total data	10010
Unique data	9392
Data with criterion: ($F_o \geq 4.0\ \sigma\ (F_o)$)	4700
$R_{sig} = \Sigma\ \sigma(F_o^2) / \Sigma\ [F_o^2]$	0.1198

c. Refinement.

Number of reflections ($F_o^2 \geq 0$)	9386
Number of refined parameters	461
Number of restraints	66
Final agreement factors:	
$wR(F^2) = [\Sigma[w(F_o^2 - F_c^2)^2] / \Sigma[w(F_o^2)^2]]^{1/2}$	0.2060
for $F_o^2 > 0$	
Weighting scheme: *a, b*	0.1077, 0.0
$w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$	
and $P = [\max(F_o^2,0) + 2F_c^2] / 3$	
$R(F) = \Sigma(\|\|F_o\| - \|F_c\|\|) / \Sigma\|F_o\|$	0.0793
for $F_o > 4.0\ \sigma\ (F_o)$	
GooF = S $=[\Sigma[w(F_o^2 - F_c^2)^2] / (n-p)]^{1/2}$	0.975
n = number of reflections	
p = number of parameters refined	
Residual electron density in final	
difference Fourier map, e/Å^3	-0.55, 0.99(10)
Max. (shift/σ) final cycle	< 0.001

Table 2a. Final fractional atomic coordinates and equivalent isotropic thermal displacement parameters with s.u.'s in parentheses.

Atoms of the Asymmetric Unit.

Atom	*x*	*y*	*z*	U_{eq} (Å^2)*
Y	-0.02685(5)	0.24702(3)	0.13405(3)	0.0290(2)
Cl	0.16296(14)	0.22907(12)	0.10852(9)	0.0593(7)
Si(1)	0.04236(18)	0.37740(12)	0.26936(10)	0.0544(8)
Si(2)	0.05049(15)	0.43712(11)	0.13092(9)	0.0426(7)
Si(3)	0.04383(17)	0.08672(12)	0.2374(1)	0.0530(8)
Si(4)	-0.17685(17)	0.08143(12)	0.19252(10)	0.0508(8)
O(1)	0.3690(4)	0.1449(3)	0.0585(3)	0.0634(19)
O(2)	0.4153(4)	0.2528(3)	0.1699(2)	0.0561(17)
O(3)	0.3652(4)	0.3172(3)	0.0341(2)	0.069(2)
N(1)	-0.1833(4)	0.2848(3)	0.0944(3)	0.0372(17)
N(2)	-0.0855(4)	0.2241(3)	0.0305(2)	0.0362(19)
C(1)	0.0028(5)	0.3686(4)	0.1865(3)	0.042(3)
C(2)	0.1809(6)	0.3519(6)	0.2788(4)	0.094(4)
C(3)	-0.0276(6)	0.3143(5)	0.3232(3)	0.071(3)
C(4)	0.0316(10)	0.4733(5)	0.3048(4)	0.122(6)
C(5)	0.1886(5)	0.4512(5)	0.1371(4)	0.062(3)
C(6)	-0.0077(6)	0.5336(4)	0.1379(3)	0.061(3)
C(7)	0.0244(5)	0.4067(4)	0.0467(3)	0.045(3)
C(8)	-0.0630(5)	0.1401(4)	0.2027(3)	0.049(3)
C(9)	0.1494(7)	0.1487(5)	0.2663(5)	0.093(4)
C(10)	0.0152(7)	0.0292(6)	0.3094(4)	0.094(4)
C(11)	0.1005(6)	0.0217(5)	0.1801(4)	0.077(4)
C(12)	-0.2517(7)	0.0814(6)	0.2659(4)	0.092(4)
C(13)	-0.1522(7)	-0.0206(5)	0.1714(5)	0.089(4)
C(14)	-0.2674(6)	0.1100(4)	0.1279(3)	0.056(3)
C(15)	-0.2689(5)	0.3249(4)	0.1217(3)	0.047(3)
C(16)	-0.2680(6)	0.3103(5)	0.1925(3)	0.062(3)
C(17)	-0.2619(6)	0.4098(4)	0.1079(4)	0.064(3)
C(18)	-0.1744(5)	0.2566(4)	0.0364(3)	0.038(2)
C(19)	-0.2523(5)	0.2598(4)	-0.0190(3)	0.053(3)
C(20)	-0.3515(6)	0.2973(6)	-0.0076(4)	0.082(4)
C(21)	-0.2038(6)	0.3021(5)	-0.0738(3)	0.070(3)
C(22)	-0.2840(6)	0.1775(5)	-0.0377(4)	0.070(3)
C(23)	-0.0473(5)	0.1825(3)	-0.0222(3)	0.036(2)
C(24)	0.0449(6)	0.2219(4)	-0.0491(3)	0.058(3)
C(25)	-0.0224(6)	0.1033(4)	-0.0027(4)	0.067(3)
C(26)	0.4365(10)	0.1322(7)	0.0114(7)	0.184(8)
C(27)	0.3915(18)	0.0780(9)	-0.0295(7)	0.348(19)
C(28)	0.3191(14)	0.0409(9)	0.0016(7)	0.21(1)
C(29)	0.3085(8)	0.0798(5)	0.0570(6)	0.113(6)
C(30)	0.4486(9)	0.1913(5)	0.2080(5)	0.098(5)
C(31)	0.4694(12)	0.2189(5)	0.2696(5)	0.139(7)
C(32)	0.4542(8)	0.2969(5)	0.2714(3)	0.089(4)
C(33)	0.4317(7)	0.3187(4)	0.2055(3)	0.073(3)
C(34)	0.3127(9)	0.3197(7)	-0.0248(4)	0.128(6)
C(35)	0.3100(11)	0.3939(7)	-0.0461(6)	0.177(8)
C(36)	0.3928(9)	0.4309(6)	-0.0146(5)	0.131(6)
C(37)	0.4138(6)	0.3873(4)	0.0424(4)	0.059(3)
Li	0.3363(9)	0.2404(7)	0.0948(5)	0.049(4)

Table 2b: Hydrogen parameters

Atom	*x*	*y*	*z*	U_{eq} (Å^2)*
H(1)	-0.06746(-)	0.38672(-)	0.18905(-)	0.05047(-)
H(2)	0.19521(-)	0.30804(-)	0.25258(-)	0.14165(-)
H(2')	0.19673(-)	0.34004(-)	0.32292(-)	0.14165(-)
H(2'')	0.22161(-)	0.39452(-)	0.26557(-)	0.14165(-)
H(3)	-0.09863(-)	0.32697(-)	0.32071(-)	0.10557(-)
H(3')	-0.0022(-)	0.32103(-)	0.36643(-)	0.10557(-)
H(3'')	-0.01848(-)	0.2618(-)	0.31043(-)	0.10557(-)
H(4)	0.06866(-)	0.50952(-)	0.27976(-)	0.18381(-)
H(4')	0.05931(-)	0.47257(-)	0.34795(-)	0.18381(-)
H(4'')	-0.03869(-)	0.48808(-)	0.30532(-)	0.18381(-)
H(5)	0.20642(-)	0.47346(-)	0.17804(-)	0.09239(-)
H(5')	0.20942(-)	0.48491(-)	0.10348(-)	0.09239(-)
H(5'')	0.22217(-)	0.40258(-)	0.13303(-)	0.09239(-)
H(6)	-0.08033(-)	0.52928(-)	0.13376(-)	0.0914(-)
H(6')	0.0167(-)	0.56624(-)	0.1045(-)	0.0914(-)
H(6'')	0.01057(-)	0.55529(-)	0.17898(-)	0.0914(-)
H(7)	0.05763(-)	0.35859(-)	0.03907(-)	0.06787(-)
H(7')	0.04977(-)	0.44483(-)	0.018(-)	0.06787(-)
H(7'')	-0.0476(-)	0.40088(-)	0.03948(-)	0.06787(-)
H(8)	-0.08217(-)	0.16869(-)	0.24098(-)	0.05887(-)
H(9)	0.17048(-)	0.18132(-)	0.23203(-)	0.13976(-)
H(9')	0.20558(-)	0.1173(-)	0.28069(-)	0.13976(-)
H(9'')	0.12704(-)	0.1797(-)	0.30132(-)	0.13976(-)
H(10)	-0.00898(-)	0.06244(-)	0.34238(-)	0.14085(-)
H(10')	0.0759(-)	0.00358(-)	0.32458(-)	0.14085(-)
H(10'')	-0.03602(-)	-0.00819(-)	0.29849(-)	0.14085(-)
H(11)	0.05211(-)	-0.01742(-)	0.16804(-)	0.11487(-)
H(11')	0.15989(-)	-0.00179(-)	0.19944(-)	0.11487(-)
H(11'')	0.11922(-)	0.04987(-)	0.14269(-)	0.11487(-)
H(12)	-0.21326(-)	0.057(-)	0.30002(-)	0.13779(-)
H(12')	-0.31405(-)	0.05393(-)	0.25806(-)	0.13779(-)
H(12'')	-0.26663(-)	0.13337(-)	0.2778(-)	0.13779(-)
H(13)	-0.12426(-)	-0.02307(-)	0.12932(-)	0.13371(-)
H(13')	-0.21483(-)	-0.04886(-)	0.17167(-)	0.13371(-)
H(13'')	-0.10478(-)	-0.04236(-)	0.20213(-)	0.13371(-)
H(14)	-0.28896(-)	0.16195(-)	0.13481(-)	0.08462(-)
H(14')	-0.32545(-)	0.07659(-)	0.12795(-)	0.08462(-)
H(14'')	-0.23503(-)	0.10642(-)	0.0873(-)	0.08462(-)
H(15)	-0.33234(-)	0.30481(-)	0.10244(-)	0.05696(-)
H(16)	-0.27183(-)	0.25609(-)	0.20026(-)	0.09365(-)
H(16')	-0.32528(-)	0.33533(-)	0.21099(-)	0.09365(-)
H(16'')	-0.20621(-)	0.33023(-)	0.21174(-)	0.09365(-)
H(17)	-0.20409(-)	0.43093(-)	0.13076(-)	0.09631(-)
H(17')	-0.32271(-)	0.43481(-)	0.12146(-)	0.09631(-)
H(17'')	-0.2546(-)	0.41747(-)	0.06263(-)	0.09631(-)
H(20)	-0.38797(-)	0.2682(-)	0.02338(-)	0.12305(-)
H(20')	-0.39062(-)	0.29974(-)	-0.04712(-)	0.12305(-)
H(20'')	-0.33996(-)	0.34839(-)	0.00843(-)	0.12305(-)
H(21)	-0.18972(-)	0.35414(-)	-0.06109(-)	0.10519(-)
H(21')	-0.24938(-)	0.30202(-)	-0.11059(-)	0.10519(-)
H(21'')	-0.1414(-)	0.2771(-)	-0.08441(-)	0.10519(-)

H(22")	-0.3182(-)	0.15396(-)	-0.00268(-)	0.10431(-)
H(23)	-0.10056(-)	0.18048(-)	-0.05601(-)	0.04256(-)
H(24)	0.02636(-)	0.27237(-)	-0.06368(-)	0.08618(-)
H(24')	0.06955(-)	0.1925(-)	-0.08437(-)	0.08618(-)
H(24")	0.09727(-)	0.22571(-)	-0.01617(-)	0.08618(-)
H(25)	0.0271(-)	0.1042(-)	0.03209(-)	0.09995(-)
H(25')	0.0048(-)	0.07606(-)	-0.03838(-)	0.09995(-)
H(25")	-0.0829(-)	0.07792(-)	0.01104(-)	0.09995(-)
H(26)	0.50028(-)	0.11293(-)	0.02951(-)	0.22088(-)
H(26')	0.44978(-)	0.17936(-)	-0.01168(-)	0.22088(-)
H(27)	0.36212(-)	0.1032(-)	-0.06713(-)	0.41935(-)
H(27')	0.44237(-)	0.0418(-)	-0.04347(-)	0.41935(-)
H(28)	0.33922(-)	-0.0118(-)	0.0101(-)	0.25309(-)
H(28')	0.25583(-)	0.04078(-)	-0.02347(-)	0.25309(-)
H(29)	0.3273(-)	0.04682(-)	0.09291(-)	0.13559(-)
H(29')	0.23782(-)	0.09441(-)	0.06121(-)	0.13559(-)
H(30)	0.39633(-)	0.15204(-)	0.20911(-)	0.11845(-)
H(30')	0.50932(-)	0.16885(-)	0.19051(-)	0.11845(-)
H(31)	0.42534(-)	0.1939(-)	0.29974(-)	0.16676(-)
H(31')	0.53933(-)	0.20732(-)	0.2821(-)	0.16676(-)
H(32)	0.51471(-)	0.323(-)	0.28773(-)	0.10657(-)
H(32')	0.3978(-)	0.30952(-)	0.29851(-)	0.10657(-)
H(33)	0.37158(-)	0.35097(-)	0.20329(-)	0.08743(-)
H(33')	0.48816(-)	0.34735(-)	0.18858(-)	0.08743(-)
H(34)	0.34649(-)	0.28746(-)	-0.0556(-)	0.15418(-)
H(34')	0.244(-)	0.30059(-)	-0.02008(-)	0.15418(-)
H(35)	0.24631(-)	0.41815(-)	-0.03522(-)	0.21274(-)
H(35')	0.31688(-)	0.39579(-)	-0.09224(-)	0.21274(-)
H(36)	0.37525(-)	0.48333(-)	-0.00392(-)	0.15753(-)
H(36')	0.45153(-)	0.4315(-)	-0.04169(-)	0.15753(-)
H(37)	0.48649(-)	0.37995(-)	0.04861(-)	0.07029(-)
H(37')	0.38818(-)	0.41378(-)	0.07957(-)	0.07029(-)

Table 3: Anisotropic (displacement) parameters (Å^2)

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Y	0.0390(3)	0.0271(3)	0.0205(3)	-0.0003(3)	-0.0067(2)	0.0059(3)
Cl	0.0428(11)	0.0760(15)	0.0589(12)	0.0055(10)	-0.0019(9)	0.0195(10)
Si(1)	0.0800(17)	0.0483(13)	0.0338(11)	-0.0055(10)	-0.0169(11)	-0.0152(12)
Si(2)	0.0555(13)	0.0357(11)	0.0366(11)	-0.0008(9)	-0.0009(9)	-0.0079(9)
Si(3)	0.0706(16)	0.0421(12)	0.0455(12)	0.0174(10)	-0.0136(11)	0.0096(11)
Si(4)	0.0630(15)	0.0433(12)	0.0464(12)	0.0127(10)	0.0050(11)	-0.0021(11)
O(1)	0.055(3)	0.052(3)	0.084(4)	-0.031(3)	0.014(3)	-0.006(3)
O(2)	0.073(3)	0.039(3)	0.055(3)	-0.003(3)	-0.020(3)	0.002(3)
O(3)	0.098(5)	0.067(4)	0.043(3)	0.011(3)	-0.004(3)	-0.027(3)
N(1)	0.043(3)	0.030(3)	0.038(3)	-0.002(3)	-0.010(3)	0.006(3)
N(2)	0.048(4)	0.037(3)	0.023(3)	-0.001(2)	-0.011(2)	0.005(3)
C(1)	0.052(5)	0.040(4)	0.034(4)	-0.011(3)	0.003(3)	-0.010(3)
C(2)	0.084(7)	0.132(9)	0.065(6)	0.040(6)	-0.033(5)	-0.041(6)
C(3)	0.088(6)	0.093(7)	0.031(4)	-0.005(4)	0.000(4)	-0.014(5)
C(4)	0.230(14)	0.078(7)	0.059(7)	-0.033(6)	0.006(8)	-0.017(8)
C(5)	0.052(5)	0.066(5)	0.066(5)	0.014(4)	-0.008(4)	-0.013(4)
C(6)	0.097(7)	0.032(4)	0.054(5)	-0.008(4)	0.010(5)	0.000(4)
C(7)	0.062(5)	0.041(4)	0.032(4)	0.005(3)	0.001(3)	0.003(4)
C(8)	0.059(5)	0.046(4)				

C(10)	0.112(8)	0.114(8)	0.056(6)	0.052(6)	0.002(5)	0.040(7)
C(11)	0.075(6)	0.072(6)	0.083(7)	0.007(5)	0.000(5)	0.025(5)
C(12)	0.089(7)	0.133(9)	0.054(6)	0.028(6)	0.011(5)	0.018(7)
C(13)	0.095(7)	0.053(6)	0.120(9)	-0.008(6)	0.001(6)	-0.002(5)
C(14)	0.066(5)	0.055(5)	0.048(5)	0.004(4)	0.002(4)	-0.010(4)
C(15)	0.040(4)	0.045(4)	0.057(5)	-0.015(4)	-0.003(3)	0.005(4)
C(16)	0.061(5)	0.074(6)	0.053(5)	-0.023(4)	0.015(4)	0.004(4)
C(17)	0.054(5)	0.047(5)	0.091(7)	0.000(5)	-0.002(5)	0.016(4)
C(18)	0.048(4)	0.037(4)	0.028(3)	-0.009(3)	-0.006(3)	-0.016(4)
C(19)	0.048(4)	0.070(6)	0.039(4)	0.009(4)	-0.011(3)	0.005(4)
C(20)	0.061(6)	0.116(8)	0.067(6)	-0.007(6)	-0.025(5)	0.014(6)
C(21)	0.080(6)	0.090(7)	0.039(5)	0.029(5)	-0.020(4)	-0.001(5)
C(22)	0.073(6)	0.085(6)	0.049(5)	-0.005(5)	-0.028(4)	-0.025(5)
C(23)	0.039(4)	0.043(4)	0.025(3)	-0.015(3)	-0.005(3)	0.008(3)
C(24)	0.070(6)	0.069(5)	0.034(4)	-0.012(4)	0.008(4)	0.010(4)
C(25)	0.094(7)	0.048(5)	0.059(5)	-0.021(4)	0.016(5)	0.022(5)
C(26)	0.169(13)	0.116(10)	0.276(19)	-0.111(12)	0.154(14)	-0.062(9)
C(27)	0.67(5)	0.151(15)	0.24(2)	-0.127(15)	0.32(3)	-0.23(2)
C(28)	0.29(2)	0.156(15)	0.184(17)	-0.099(14)	-0.017(16)	-0.098(16)
C(29)	0.121(9)	0.048(6)	0.174(13)	-0.005(7)	0.069(9)	0.003(6)
C(30)	0.152(10)	0.044(5)	0.097(8)	0.001(6)	-0.035(7)	0.021(6)
C(31)	0.285(19)	0.054(6)	0.077(8)	0.020(6)	-0.024(10)	0.019(9)
C(32)	0.151(10)	0.063(6)	0.050(6)	-0.007(5)	-0.042(6)	0.022(6)
C(33)	0.110(7)	0.050(5)	0.057(6)	-0.005(4)	-0.034(5)	0.005(5)
C(34)	0.176(12)	0.159(12)	0.048(6)	0.026(7)	-0.035(7)	-0.092(10)
C(35)	0.203(15)	0.181(14)	0.141(12)	0.102(11)	-0.112(11)	-0.097(12)
C(36)	0.158(12)	0.126(10)	0.106(9)	0.066(8)	-0.051(8)	-0.061(9)
C(37)	0.059(5)	0.051(5)	0.067(6)	0.004(4)	0.008(4)	-0.002(4)
Li	0.055(7)	0.055(8)	0.038(6)	-0.013(6)	-0.005(5)	0.005(6)

Table 4. Selected data on the geometry.

Standard deviations in the last decimal place are given in parentheses.

Interatomic Distances (Å)

Y	-Cl	2.639(2)	O(2)	-C(33)	1.409(9)
Y	-N(1)	2.339(6)	O(2)	-Li	1.905(12)
Y	-N(2)	2.353(4)	O(3)	-C(34)	1.421(10)
Y	-C(1)	2.460(7)	O(3)	-C(37)	1.416(9)
Y	-C(8)	2.454(7)	O(3)	-Li	1.926(13)
Y	-C(18)	2.834(7)	N(1)	-C(15)	1.484(9)
Cl	-Li	2.362(12)	N(1)	-C(18)	1.340(9)
Si(1)	-C(1)	1.833(7)	N(2)	-C(18)	1.335(9)
Si(1)	-C(2)	1.919(9)	N(2)	-C(23)	1.449(8)
Si(1)	-C(3)	1.874(8)	C(15)	-C(16)	1.528(9)
Si(1)	-C(4)	1.872(9)	C(15)	-C(17)	1.541(10)
Si(2)	-C(1)	1.826(7)	C(18)	-C(19)	1.554(9)
Si(2)	-C(5)	1.871(7)	C(19)	-C(20)	1.515(11)
Si(2)	-C(6)	1.893(8)	C(19)	-C(21)	1.546(10)
Si(2)	-C(7)	1.894(7)	C(19)	-C(22)	1.572(11)
Si(3)	-C(8)	1.855(7)	C(23)	-C(24)	1.546(10)
Si(3)	-C(9)	1.884(10)	C(23)	-C(25)	1.503(9)
Si(3)	-C(10)	1.890(9)	C(26)	-C(27)	1.42(2)

Si(4)	-C(12)	1.880(9)
Si(4)	-C(13)	1.900(9)
Si(4)	-C(14)	1.879(7)
O(1)	-C(26)	1.388(15)
O(1)	-C(29)	1.414(11)
O(1)	-Li	1.921(13)
O(2)	-C(30)	1.425(11)
C(30)	-C(31)	1.419(15)
C(31)	-C(32)	1.403(13)
C(32)	-C(33)	1.477(9)
C(34)	-C(35)	1.395(17)
C(35)	-C(36)	1.440(18)
C(36)	-C(37)	1.460(13)

Bond angles (deg.)

Cl	-Y	-N(1)	145.33(16)
Cl	-Y	-N(2)	94.92(14)
Cl	-Y	-C(1)	93.14(16)
Cl	-Y	-C(8)	103.71(17)
Cl	-Y	-C(18)	121.00(14)
N(1)	-Y	-N(2)	55.9(2)
N(1)	-Y	-C(1)	92.5(2)
N(1)	-Y	-C(8)	104.3(2)
N(1)	-Y	-C(18)	28.0(2)
N(2)	-Y	-C(1)	128.5(2)
N(2)	-Y	-C(8)	110.9(2)
N(2)	-Y	-C(18)	27.90(19)
C(1)	-Y	-C(8)	116.3(2)
C(1)	-Y	-C(18)	112.3(2)
C(8)	-Y	-C(18)	109.7(2)
Y	-Cl	-Li	167.2(3)
C(1)	-Si(1)	-C(2)	109.5(4)
C(1)	-Si(1)	-C(3)	113.6(3)
C(1)	-Si(1)	-C(4)	116.2(4)
C(2)	-Si(1)	-C(3)	107.2(4)
C(2)	-Si(1)	-C(4)	105.0(5)
C(3)	-Si(1)	-C(4)	104.7(4)
C(1)	-Si(2)	-C(5)	114.1(4)
C(1)	-Si(2)	-C(6)	113.6(3)
C(1)	-Si(2)	-C(7)	111.3(3)
C(5)	-Si(2)	-C(6)	106.5(4)
C(5)	-Si(2)	-C(7)	105.1(3)
C(6)	-Si(2)	-C(7)	105.5(3)
C(8)	-Si(3)	-C(9)	113.3(4)
C(8)	-Si(3)	-C(10)	115.3(4)
C(8)	-Si(3)	-C(11)	112.6(3)
C(9)	-Si(3)	-C(10)	102.7(4)
C(9)	-Si(3)	-C(11)	105.1(4)
C(10)	-Si(3)	-C(11)	106.9(4)
C(8)	-Si(4)	-C(12)	111.1(4)
C(8)	-Si(4)	-C(13)	114.6(4)
C(8)	-Si(4)	-C(14)	116.5(3)
C(12)	-Si(4)	-C(13)	107.3(5)
C(12)	-Si(4)	-C(14)	104.9(4)
C(13)	-Si(4)	-C(14)	101.5(4)
C(26)	-O(1)	-C(29)	103.7(8)
C(26)	-O(1)	-Li	126.5(7)
C(29)	-O(1)	-Li	126.5(7)
C(30)	-O(2)	-C(33)	106.9(6)
Y	-N(1)	-C(15)	133.9(4)
Y	-N(1)	-C(18)	97.0(4)
C(15)	-N(1)	-C(18)	129.0(6)
Y	-N(2)	-C(18)	96.5(4)
Y	-N(2)	-C(23)	134.1(4)
C(18)	-N(2)	-C(23)	129.1(5)
Y	-C(1)	-Si(1)	123.4(3)
Y	-C(1)	-Si(2)	110.4(3)
Si(1)	-C(1)	-Si(2)	117.9(4)
Y	-C(8)	-Si(3)	118.0(3)
Y	-C(8)	-Si(4)	122.8(3)
Si(3)	-C(8)	-Si(4)	112.5(4)
N(1)	-C(15)	-C(16)	108.6(6)
N(1)	-C(15)	-C(17)	110.1(6)
C(16)	-C(15)	-C(17)	110.8(6)
Y	-C(18)	-N(1)	55.0(3)
Y	-C(18)	-N(2)	55.6(3)
Y	-C(18)	-C(19)	177.5(5)
N(1)	-C(18)	-N(2)	110.6(5)
N(1)	-C(18)	-C(19)	127.5(6)
N(2)	-C(18)	-C(19)	121.9(6)
C(18)	-C(19)	-C(20)	118.1(6)
C(18)	-C(19)	-C(21)	107.5(6)
C(18)	-C(19)	-C(22)	109.2(6)
C(20)	-C(19)	-C(21)	107.3(6)
C(20)	-C(19)	-C(22)	102.6(6)
C(21)	-C(19)	-C(22)	112.3(6)
N(2)	-C(23)	-C(24)	111.1(5)
N(2)	-C(23)	-C(25)	110.2(5)
C(24)	-C(23)	-C(25)	110.7(6)
O(1)	-C(26)	-C(27)	106.1(12)
C(26)	-C(27)	-C(28)	109.1(14)
C(27)	-C(28)	-C(29)	105.4(14)
O(1)	-C(29)	-C(28)	110.9(11)
O(2)	-C(30)	-C(31)	108.0(7)
C(30)	-C(31)	-C(32)	110.0(8)
C(31)	-C(32)	-C(33)	105.0(7)
O(2)	-C(33)	-C(32)	108.4(6)
O(3)	-C(34)	-C(35)	108.9(10)
C(34)	-C(35)	-C(36)	105.6(11)
C(35)	-C(36)	-C(37)	105.8(9)
O(3)	-C(37)	-C(36)	106.7(7)
Cl	-Li	-O(1)	102.2(5)
Cl	-Li	-O(2)	115.7(5)

C(34)	-O(3)	-C(37)	107.3(7)	O(1)	-Li	-O(3)	107.7(6)
C(34)	-O(3)	-Li	120.6(7)	O(2)	-Li	-O(3)	111.2(6)
C(37)	-O(3)	-Li	129.9(6)				

Part II: Structure determination of $[PhC(NSiMe_3)(CH_2)_2NMe_2]Y[CH(SiMe_3)_2]_2$ (2a).

Abstract. $C_{28}H_{62}N_3Si_5Y$, M = 670.15, triclinic, P , a = 9.350(5), b = 10.724(1), c = 20.363(5) Å, α = 88.71(1)°, β = 81.68(3)°, γ = 75.09(2)°, V = 1952.0(12) Å^3, Z = 2, D_x = 1.140 g cm^{-3}, λ(MoK) = 0.71073 Å, μ = 16.67 cm^{-1}, F(000) = 720, T = 130 K, $wR(F^2)$ = 0.1441 for 8465 reflections with $F_o^2 \geq 0$ and 582 parameters and $R(F)$ = 0.0488 for 6480 reflections obeying $F_o \geq 4.0\ \sigma(F_o)$ criterion of observability. The asymmetric unit consists of one molecule of the title compound.

Experimental

X-ray diffraction: Crystal and Molecular Structure.

Suitable light yellow colored crystals were obtained by recrystallisation from pentane.

The crystal, a parallelepiped of approximate size 0.25 x 0.30 x 0.33 mm., used for characterization and data collection was glued on top of a glass fiber by using inert-atmosphere handling techniques and was transferred into the cold nitrogen cold stream of the low temperature unit[1] mounted on an Enraf-Nonius *CAD-4F*[2] diffractometer, interfaced to a *INDY* (*Silicon Graphics*) *UNIX* computer (Mo tube, 50 kV, 40 mA, monochromated Mo-K radiation, $\Delta\omega = 1.15 + 0.34$ tg θ). Reflections profiles showed large mosaicity; this mosaicity did not allow to use a narrower scan angle.

Unit cell parameters[3] and orientation matrix were determined from a least-squares treatment of the *SET4*[4] setting angles of 22 reflections in the range 16.79° < θ < 21.62°. The unit cell was identified as triclinic, space group P . Reduced cell calculations did not indicate any higher metric lattice symmetry[5] and examination of the final atomic coordinates of the structure did not yield extra metric symmetry elements.[6,7]

The intensities of three standard reflections, monitored every three hours of X-ray exposure time, showed no greater fluctuations during data collection than those expected from Poisson statistics. A 360° ψ-scan for a reflection close to axial (-2-1-3) showed a variation in intensity of less than 9% about the mean value. Intensity data were corrected for Lorentz and polarization effects, scale variation, but not for absorption and reduced to F_o^2.[8]

The structure was solved by Patterson methods and extension of the model was accomplished by direct methods applied to difference structure factors using the program *DIRDIF*.[9] The positional and anisotropic thermal displacement parameters for the non-hydrogen atoms were refined. A subsequent difference Fourier synthesis resulted in the location of all the hydrogen atoms, which coordinates and isotropic thermal displacement parameters were refined.

Final refinement on F^2 carried out by full-matrix least-squares techniques converged at $wR(F^2)$ = 0.1441 for 8465 reflections with $F_o^2 \geq 0$ and $R(F)$ = 0.0488 for 6480 reflections with $F_o \geq 4.0\ \sigma(F_o)$ and 582 parameters. The final difference Fourier map was essentially featureless with a few peaks of max. 1.08(12) e/Å^3 within 1.0 Å from Y, but were neglected/rejected, being artefacts. No other significant

peaks having chemical meaning above the general background were observed in the final difference Fourier syntheses.

The positional and anisotropic thermal displacement parameters for the non-hydrogen atoms and isotropic thermal displacement parameters for hydrogen atoms were refined on F^2 with full-matrix least-squares procedures minimizing the function $Q = \Sigma_h [w(|F_o^2 - kF_c^2|)^2]$, where $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$, $P = [\max(F_o^2,0) + 2F_c^2] / 3$, F_o and F_c are the observed and calculated structure factor amplitudes, respectively; a and b were refined. Reflections were stated observed if satisfying $F^2 > 0$ criterion of observability.

Neutral atom scattering factors and anomalous dispersion corrections were taken from *International Tables of Crystallography.*[10] All calculations performed on the HP9000/735 computer at the University of Groningen with the program packages *SHELXL*[11] (least-square refinements), *PLATON*[12] (calculation of geometric data and the *ORTEP* illustrations) and a locally modified version of the program *PLUTO*[15] (preparation of illustrations).

The triclinic unit cell contains two discrete units of each type of the title compound separated by normal van der Waals distances[16] (Fig.).

No missed symmetry (*MISSYM*) detected by procedures implemented in *PLATON.*[17]

The structure contains small potential residual solvent accessible voids.[18]

Table 1.

a. Crystal data and details of the structure determination.

Moiety_ Formula	$C_{28}H_{62}N_3Si_5Y$
Formula_Weight, g.mol^{-1}	670.15
Crystal system	triclinic
Space group,	*P*
a, Å	9.350(5)
b, Å	10.724(1)
c, Å	20.363(5)
α, deg	88.71(1)
β, deg	81.68(3)
γ, deg	75.09(2)
V, Å^3	1952.0(12)
Formula_*Z*	2
SpaceGroup_*Z*	2
Z' (= Formula_Z / SpaceGroup-*Z*)	1
ρ_{calc}, g.cm^{-3}	1.140
F(000), electrons	720
μ(Mo K), cm^{-1}	16.67
color, habit	light yellow, parallelepiped
Approx. crystal dimension, mm	0.25 x 0.30 x 0.35

b. Data collection.

Radiation	Mo K
Wavelength, Å	0.71073
Monochromator	Graphite
Temperature, K	130
θ range; min. max., deg	1.01, 27.0
ω/2θ scan, deg	$\Delta\omega = 1.15 + 0.34 \text{ tg } \theta$
Index ranges	h: -11→11; k: -13→13; l: -25→0
Crystal-to-receiving-aperture-distance, mm	173
Horizontal-, vertical-aperture, mm	3.2 + tg θ; 4.0
Reference reflections,	-114, 2.2
r.m.s. dev. in %	0-2-1, 0.8
	-2-10, 3.1
Drift correction	0.984 - 1.000
X-ray exposure time, h	181.0
Total data	8709
Unique data	8465
Data with criterion: ($F_o \geq 4.0\ \sigma\ (F_o)$)	6480
$R_{int} = \Sigma\ [\lvert F_o^2 - F_o^2\text{(mean)}\rvert] / \Sigma\ [F_o^2]$	0.021
$R_{sig} = \Sigma\ \sigma(F_o^2) / \Sigma\ [F_o^2]$	0.048

c. Refinement.

Number of reflections ($F_o^2 \geq 0$)	8465
Number of refined parameters	2582
Final agreement factors:	
$wR(F^2) = [\Sigma[w(F_o^2 - F_c^2)^2] / \Sigma[w(F_o^2)^2]]^{1/2}$	0.1441
for $F_o^2 > 0$	
Weighting scheme: *a, b*	0.0782, 2.57
$w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$	
and $P = [\max(F_o^2,0) + 2F_c^2] / 3$	
$R(F) = \Sigma(\|\|F_o\| - \|F_c\|\|) / \Sigma\|F_o\|$	0.0488
for $F_o > 4.0\ \sigma\ (F_o)$	
GooF = S $=[\Sigma[w(F_o^2 - F_c^2)^2] / (n-p)]^{1/2}$	1.058
n = number of reflections	
p = number of parameters refined	
Residual electron density in final	
difference Fourier map, e/Å^3	-1.76, 1.08(12)
Max. (shift/σ) final cycle	< 0.001

Table 2a. Final fractional atomic coordinates and equivalent isotropic thermal displacement parameters for non-H atoms with s.u.'s in parentheses.

Atoms of the Asymmetric Unit.

Atom	*x*	*y*	*z*	U_{eq} (Å^2)*
Y(1)	0.23083(4)	0.05771(3)	0.26674(2)	0.0176(1)
Si(1)	0.29393(14)	0.33127(11)	0.14932(6)	0.0281(4)
Si(3)	0.53847(13)	-0.16322(11)	0.15861(6)	0.0289(3)
Si(4)	0.49236(13)	-0.23388(11)	0.30836(6)	0.0279(3)
Si(5)	0.34119(13)	0.20950(11)	0.41214(5)	0.0261(3)
Si(6)	0.01931(13)	0.32957(11)	0.37860(6)	0.0298(3)
N(1)	0.2214(4)	0.2032(3)	0.17740(15)	0.0218(9)
N(2)	0.1013(4)	0.0461(3)	0.18117(16)	0.0263(10)
N(3)	0.0177(4)	-0.0541(3)	0.29768(16)	0.0244(10)
C(1)	0.1183(5)	0.1559(4)	0.15396(18)	0.0230(11)
C(2)	0.0291(5)	0.2163(4)	0.10092(19)	0.0265(11)
C(3)	-0.0794(6)	0.3319(5)	0.1110(3)	0.0393(16)
C(4)	-0.1628(8)	0.3822(6)	0.0611(3)	0.055(2)
C(5)	-0.1398(8)	0.3197(6)	0.0016(3)	0.061(2)
C(6)	-0.0285(8)	0.2043(6)	-0.0096(3)	0.060(2)
C(7)	0.0523(7)	0.1523(5)	0.0407(2)	0.0434(18)
C(8)	0.4705(7)	0.3091(6)	0.1851(3)	0.0423(17)
C(9)	0.1678(7)	0.4895(5)	0.1784(3)	0.0420(19)
C(10)	0.3370(9)	0.3346(6)	0.0569(3)	0.0476(18)
C(11)	0.4650(5)	-0.1072(4)	0.2447(2)	0.0239(11)
C(12)	0.4114(7)	-0.2439(7)	0.1229(3)	0.0492(19)
C(13)	0.5626(8)	-0.0275(6)	0.1027(3)	0.0464(19)
C(14)	0.7288(6)	-0.2807(6)	0.1481(4)	0.0501(19)
C(15)	0.4621(7)	-0.3901(5)	0.2821(3)	0.045(2)
C(16)	0.6848(6)	-0.2778(5)	0.3327(3)	0.0453(19)
C(17)	0.3670(8)	-0.1816(7)	0.3894(3)	0.0449(17)
C(18)	0.2078(5)	0.2242(4)	0.35233(19)	0.0234(11)
C(19)	0.5348(5)	0.1210(5)	0.3748(3)	0.0381(16)
C(20)	0.2865(7)	0.1249(6)	0.4896(3)	0.0482(18)
C(21)	0.3678(9)	0.3647(6)	0.4426(4)	0.054(2)
C(22)	-0.1034(7)	0.3365(6)	0.3120(3)	0.0406(17)
C(23)	0.0169(9)	0.5012(6)	0.3943(5)	0.056(2)
C(24)	-0.0781(7)	0.2779(8)	0.4564(3)	0.052(2)
C(25)	-0.0279(5)	-0.0043(5)	0.1811(2)	0.0317(12)
C(26)	-0.0986(5)	-0.0068(4)	0.2530(2)	0.0273(12)
C(27)	-0.0561(6)	-0.0389(6)	0.3665(2)	0.0373(14)
C(28)	0.0872(6)	-0.1918(4)	0.2818(3)	0.0334(14)

*) $U_{eq} = 1/3\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$ [19]

Table 2b: Hydrogen parameters

Atom	*x*	*y*	*z*	U_{eq} (Å^2)*
H(3)	-0.097(5)	0.378(5)	0.146(2)	0.034(13)
H(4)	-0.233(6)	0.452(6)	0.069(3)	0.049(16)
H(5)	-0.197(5)	0.361(5)	-0.042(2)	0.041(14)
H(6)	-0.015(7)	0.161(7)	-0.051(3)	0.08(2)
H(7)	0.124(6)	0.087(5)	0.037(2)	0.032(14)
H(8)	0.438(8)	0.317(7)	0.226(4)	0.08(2)
H(8')	0.505(12)	0.377(12)	0.187(6)	0.17(5)
H(8'')	0.533(7)	0.232(7)	0.175(3)	0.064(19)
H(9)	0.072(7)	0.509(5)	0.167(3)	0.049(16)
H(9')	0.218(5)	0.558(5)	0.175(2)	0.035(13)
H(9'')	0.141(4)	0.491(4)	0.217(2)	0.01(1)
H(10)	0.394(7)	0.261(7)	0.046(3)	0.059(19)
H(10')	0.389(6)	0.395(6)	0.049(3)	0.051(16)
H(10'')	0.243(7)	0.358(6)	0.043(3)	0.059(19)
H(11)	0.520(5)	-0.055(4)	0.262(2)	0.030(12)
H(12)	0.395(7)	-0.325(7)	0.149(3)	0.07(2)
H(12')	0.452(5)	-0.273(5)	0.082(3)	0.032(13)
H(12'')	0.325(7)	-0.183(6)	0.122(3)	0.06(2)
H(13)	0.472(6)	0.035(5)	0.103(3)	0.045(15)
H(13')	0.599(6)	-0.061(5)	0.059(3)	0.043(14)
H(13'')	0.635(7)	0.010(6)	0.106(3)	0.07(2)
H(14)	0.790(9)	-0.239(8)	0.172(4)	0.11(3)
H(14')	0.769(7)	-0.298(7)	0.106(3)	0.07(2)
H(14'')	0.719(7)	-0.353(6)	0.184(3)	0.065(18)
H(15)	0.364(9)	-0.392(8)	0.274(4)	0.10(3)
H(15')	0.464(7)	-0.437(6)	0.308(3)	0.05(2)
H(15'')	0.541(8)	-0.436(7)	0.238(4)	0.09(2)
H(16)	0.749(6)	-0.311(5)	0.295(2)	0.031(13)
H(16')	0.684(6)	-0.344(6)	0.375(3)	0.058(17)
H(16'')	0.706(8)	-0.202(8)	0.351(4)	0.09(2)
H(17)	0.417(8)	-0.148(7)	0.424(4)	0.08(2)
H(17')	0.325(9)	-0.240(8)	0.415(4)	0.09(2)
H(17'')	0.301(13)	-0.137(12)	0.399(6)	0.15(5)
H(18)	0.250(5)	0.268(4)	0.312(2)	0.021(10)
H(19)	0.540(6)	0.043(6)	0.365(3)	0.042(15)
H(19')	0.606(6)	0.122(5)	0.409(3)	0.053(16)
H(19'')	0.570(7)	0.167(7)	0.329(3)	0.08(2)
H(20)	0.203(8)	0.173(6)	0.518(3)	0.067(19)
H(20')	0.355(12)	0.126(11)	0.532(6)	0.18(4)
H(20'')	0.281(11)	0.037(10)	0.465(5)	0.14(4)
H(21)	0.377(7)	0.418(6)	0.405(3)	0.057(18)
H(21')	0.448(9)	0.354(7)	0.453(4)	0.08(3)
H(21'')	0.297(7)	0.413(6)	0.459(3)	0.047(18)
H(22)	-0.065(8)	0.372(7)	0.285(3)	0.06(2)
H(22')	-0.191(7)	0.396(6)	0.308(3)	0.055(17)
H(22'')	-0.114(6)	0.259(5)	0.300(2)	0.039(14)
H(23)	0.069(5)	0.510(4)	0.427(2)	0.028(12)
H(23')	-0.079(7)	0.555(6)	0.413(3)	0.063(18)
H(23'')	0.048(7)	0.515(6)	0.363(3)	0.04(2)
H(24)	-0.071(6)	0.201(6)	0.456(3)	0.038(17)
H(24')	-0.179(8)	0.322(6)	0.461(3)	0.067(19)
H(24'')	-0.044(6)	0.301(5)	0.495(3)	0.052(16)
H(25)	-0.002(6)	0.087(6)		

H(26)	-0.156(6)	0.076(5)	0.271(2)	0.040(14)
H(26')	-0.168(5)	-0.059(4)	0.258(2)	0.024(11)
H(27)	-0.131(6)	-0.086(5)	0.380(3)	0.053(16)
H(27')	-0.103(5)	0.053(5)	0.380(2)	0.029(12)
H(27'')	0.018(5)	-0.061(4)	0.398(2)	0.023(11)
H(28)	0.155(4)	-0.229(4)	0.3123(19)	0.011(9)
H(28')	0.152(5)	-0.204(4)	0.239(2)	0.028(12)
H(28'')	0.013(5)	-0.243(4)	0.280(2)	0.023(11)

Table 3: Anisotropic (displacement) parameters (Å^2)

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Y(1)	0.0197(2)	0.0150(2)	0.0183(2)	0.0013(1)	-0.0043(1)	-0.0041(1)
Si(1)	0.0404(7)	0.0210(5)	0.0263(6)	0.0032(4)	-0.0030(5)	-0.0151(5)
Si(3)	0.0269(6)	0.0252(6)	0.0333(6)	-0.0084(5)	0.0010(5)	-0.0068(5)
Si(4)	0.0278(6)	0.0204(5)	0.0374(7)	0.0029(5)	-0.0131(5)	-0.0054(4)
Si(5)	0.0291(6)	0.0249(6)	0.0222(5)	-0.0013(4)	-0.0080(4)	-0.0006(5)
Si(6)	0.0300(6)	0.0262(6)	0.0288(6)	-0.0089(5)	-0.0096(5)	0.0046(5)
N(1)	0.0339(18)	0.0198(16)	0.0144(15)	0.0006(12)	-0.0056(13)	-0.0108(14)
N(2)	0.0342(19)	0.0253(18)	0.0248(17)	0.0076(14)	-0.0083(14)	-0.0158(15)
N(3)	0.0235(17)	0.0266(17)	0.0238(17)	0.0048(14)	-0.0037(13)	-0.0077(14)
C(1)	0.030(2)	0.0225(19)	0.0180(18)	0.0010(15)	-0.0047(16)	-0.0086(16)
C(2)	0.038(2)	0.028(2)	0.0187(19)	0.0057(16)	-0.0122(17)	-0.0136(18)
C(3)	0.058(3)	0.027(2)	0.035(3)	-0.003(2)	-0.022(2)	-0.006(2)
C(4)	0.077(4)	0.034(3)	0.060(4)	0.010(3)	-0.044(3)	-0.006(3)
C(5)	0.102(5)	0.045(3)	0.049(3)	0.016(3)	-0.052(3)	-0.023(3)
C(6)	0.106(5)	0.055(4)	0.029(3)	0.004(3)	-0.034(3)	-0.024(4)
C(7)	0.067(4)	0.039(3)	0.023(2)	-0.003(2)	-0.014(2)	-0.007(3)
C(8)	0.043(3)	0.039(3)	0.050(3)	-0.001(2)	-0.005(2)	-0.020(2)
C(9)	0.058(4)	0.028(3)	0.041(3)	-0.001(2)	-0.001(3)	-0.016(2)
C(10)	0.076(4)	0.042(3)	0.027(2)	0.008(2)	0.002(3)	-0.025(3)
C(11)	0.024(2)	0.0172(18)	0.031(2)	-0.0020(16)	-0.0070(16)	-0.0041(16)
C(12)	0.048(3)	0.064(4)	0.040(3)	-0.023(3)	0.004(3)	-0.026(3)
C(13)	0.055(4)	0.039(3)	0.038(3)	-0.003(2)	0.010(3)	-0.008(3)
C(14)	0.038(3)	0.039(3)	0.064(4)	-0.017(3)	0.010(3)	-0.001(2)
C(15)	0.056(4)	0.022(2)	0.061(4)	0.007(2)	-0.021(3)	-0.013(2)
C(16)	0.037(3)	0.033(3)	0.069(4)	0.008(3)	-0.026(3)	-0.005(2)
C(17)	0.049(3)	0.054(3)	0.038(3)	0.004(3)	-0.010(2)	-0.023(3)
C(18)	0.030(2)	0.0178(18)	0.0199(19)	0.0022(15)	-0.0040(16)	-0.0017(16)
C(19)	0.030(2)	0.039(3)	0.041(3)	-0.004(2)	-0.010(2)	0.002(2)
C(20)	0.052(3)	0.063(4)	0.021(2)	0.011(2)	-0.006(2)	0.000(3)
C(21)	0.055(4)	0.040(3)	0.069(4)	-0.018(3)	-0.031(3)	-0.002(3)
C(22)	0.042(3)	0.034(3)	0.045(3)	-0.006(2)	-0.022(3)	0.001(2)
C(23)	0.060(4)	0.035(3)	0.067(5)	-0.023(3)	-0.035(4)	0.016(3)
C(24)	0.047(4)	0.072(5)	0.032(3)	-0.011(3)	0.001(2)	-0.007(3)
C(25)	0.037(2)	0.032(2)	0.036(2)	0.008(2)	-0.018(2)	-0.020(2)
C(26)	0.023(2)	0.029(2)	0.034(2)	0.0035(18)	-0.0085(17)	-0.0118(18)
C(27)	0.034(2)	0.058(3)	0.021(2)	0.004(2)	-0.0007(19)	-0.016(2)
C(28)	0.034(2)	0.027(2)	0.042(3)	0.007(2)	-0.010(2)	-0.0110(19)

Table 4. Selected data on the geometry.

Standard deviations in the last decimal place are given in parentheses.

Interatomic Distances (Å)

Y(1)	-N(1)	2.366(3)	Si(5)	-C(20)	1.874(6)
Y(1)	-N(2)	2.284(4)	Si(5)	-C(21)	1.876(7)
Y(1)	-N(3)	2.576(4)	Si(6)	-C(18)	1.844(5)
Y(1)	-C(1)	2.740(4)	Si(6)	-C(22)	1.888(7)
Y(1)	-C(11)	2.425(5)	Si(6)	-C(23)	1.869(7)
Y(1)	-C(18)	2.471(4)	Si(6)	-C(24)	1.857(7)
Si(1)	-N(1)	1.733(4)	N(1)	-C(1)	1.345(6)
Si(1)	-C(8)	1.858(7)	N(2)	-C(1)	1.327(5)
Si(1)	-C(9)	1.853(6)	N(2)	-C(25)	1.445(6)
Si(1)	-C(10)	1.868(6)	N(3)	-C(26)	1.500(6)
Si(3)	-C(11)	1.842(4)	N(3)	-C(27)	1.463(5)
Si(3)	-C(12)	1.870(7)	N(3)	-C(28)	1.475(5)
Si(3)	-C(13)	1.867(6)	C(1)	-C(2)	1.494(6)
Si(3)	-C(14)	1.884(6)	C(2)	-C(3)	1.383(7)
Si(4)	-C(11)	1.852(4)	C(2)	-C(7)	1.381(6)
Si(4)	-C(15)	1.871(6)	C(3)	-C(4)	1.386(9)
Si(4)	-C(16)	1.875(6)	C(4)	-C(5)	1.360(9)
Si(4)	-C(17)	1.886(7)	C(5)	-C(6)	1.396(9)
Si(5)	-C(18)	1.842(5)	C(6)	-C(7)	1.383(8)
Si(5)	-C(19)	1.872(5)	C(25)	-C(26)	1.519(6)

Bond angles (deg.)

N(1)	-Y(1)	-N(2)	57.75(12)	C(18)	-Si(6)	-C(23)	113.9(3)
N(1)	-Y(1)	-N(3)	119.76(12)	C(18)	-Si(6)	-C(24)	113.6(3)
N(1)	-Y(1)	-C(1)	29.40(13)	C(22)	-Si(6)	-C(23)	105.1(3)
N(1)	-Y(1)	-C(11)	107.55(13)	C(22)	-Si(6)	-C(24)	107.7(3)
N(1)	-Y(1)	-C(18)	94.61(12)	C(23)	-Si(6)	-C(24)	105.1(4)
N(2)	-Y(1)	-N(3)	66.57(12)	Y(1)	-N(1)	-Si(1)	139.5(2)
N(2)	-Y(1)	-C(1)	28.83(13)	Y(1)	-N(1)	-C(1)	90.9(2)
N(2)	-Y(1)	-C(11)	106.48(14)	Si(1)	-N(1)	-C(1)	129.5(3)
N(2)	-Y(1)	-C(18)	133.35(14)	Y(1)	-N(2)	-C(1)	95.0(3)
N(3)	-Y(1)	-C(1)	91.50(13)	Y(1)	-N(2)	-C(25)	128.8(3)
N(3)	-Y(1)	-C(11)	108.46(13)	C(1)	-N(2)	-C(25)	125.5(4)
N(3)	-Y(1)	-C(18)	108.37(13)	Y(1)	-N(3)	-C(26)	109.0(2)
C(1)	-Y(1)	-C(11)	113.42(13)	Y(1)	-N(3)	-C(27)	116.6(3)
C(1)	-Y(1)	-C(18)	113.34(13)	Y(1)	-N(3)	-C(28)	104.8(3)
C(11)	-Y(1)	-C(18)	118.17(15)	C(26)	-N(3)	-C(27)	108.5(4)
N(1)	-Si(1)	-C(8)	105.8(2)	C(26)	-N(3)	-C(28)	107.7(3)
N(1)	-Si(1)	-C(9)	112.4(2)	C(27)	-N(3)	-C(28)	110.0(4)
N(1)	-Si(1)	-C(10)	113.1(2)	Y(1)	-C(1)	-N(1)	59.7(2)
C(8)	-Si(1)	-C(9)	108.4(3)	Y(1)	-C(1)	-N(2)	56.1(2)
C(8)	-Si(1)	-C(10)	108.8(3)	Y(1)	-C(1)	-C(2)	169.1(3)
C(9)	-Si(1)	-C(10)	108.2(3)	N(1)	-C(1)	-N(2)	114.5(4)
C(11)	-Si(3)	-C(12)	111.9(3)	N(1)	-C(1)	-C(2)	125.0(4)
C(11)	-Si(3)	-C(13)	112.0(2)	N(2)	-C(1)	-C(2)	120.6(4)
C(11)	-Si(3)	-C(14)	114.5(3)	C(1)	-C(2)	-C(3)	122.1(4)
C(12)	-Si(3)	-C(13)	107.1(3)	C(1)	-C(2)	-C(7)	118.8(4)
C(12)	-Si(3)	-C(14)	106.6(3)	C(3)	-C(2)	-C(7)	119.0(5)

C(11)	-Si(4)	-C(16)	113.9(2)	C(4)	-C(5)	-C(6)	119.3(6)
C(11)	-Si(4)	-C(17)	112.4(3)	C(5)	-C(6)	-C(7)	119.7(5)
C(15)	-Si(4)	-C(16)	104.8(3)	C(2)	-C(7)	-C(6)	120.8(5)
C(15)	-Si(4)	-C(17)	107.7(3)	Y(1)	-C(11)	-Si(3)	119.2(2)
C(16)	-Si(4)	-C(17)	103.4(3)	Y(1)	-C(11)	-Si(4)	114.4(2)
C(18)	-Si(5)	-C(19)	111.5(2)	Si(3)	-C(11)	-Si(4)	115.3(2)
C(18)	-Si(5)	-C(20)	112.5(3)	Y(1)	-C(18)	-Si(5)	122.9(2)
C(18)	-Si(5)	-C(21)	116.2(3)	Y(1)	-C(18)	-Si(6)	116.9(2)
C(19)	-Si(5)	-C(20)	108.4(3)	Si(5)	-C(18)	-Si(6)	115.8(2)
C(19)	-Si(5)	-C(21)	103.0(3)	N(2)	-C(25)	-C(26)	106.9(3)
C(20)	-Si(5)	-C(21)	104.5(3)	N(3)	-C(26)	-C(25)	111.4(4)
C(18)	-Si(6)	-C(22)	110.8(2)				

Part III: Structure determination of $Li[PhC(NSiMe_3)(CH_2)_2NMe_2]_2Y(CH_2Ph)_2$ (3a).

Abstract. $C_{42}H_{62}LiN_6Si_2Y$, M = 803.01, monoclinic, $P2_1/n$, a = 19.695(3), b = 19.968(3), c = 23.502(3) Å, β = 101.37(1)°, V = 9061(2) Å^3, Z = 8, D_x = 1.177 g cm^{-3}, λ(MoK) = 0.71073 Å, μ = 13.7 cm^{-1}, F(000) = 3408 T = 130 K, $wR(F^2)$ = 0.1998 for 15914 reflections with $F_o^2 \geq 0$ and 957 parameters and $R(F)$ = 0.0840 for 7483 reflections obeying $F_o \geq 4.0\ \sigma(F_o)$ criterion of observability.

The asymmetric unit consists of two molecules of the title compound.

Experimental

X-ray diffraction: Crystal and Molecular Structure.

Suitable light-yellow colored block-shaped crystals were obtained by recrystallisation from toluene. The crystal, a parallelepiped of approximate size 0.15 x 0.19 x 0.28 mm., used for characterization and data collection was glued on top of a glass fiber by using inert-atmosphere handling techniques and was transferred into the cold nitrogen cold stream of the low temperature unit[1] mounted on an Enraf-Nonius *CAD-4F*^{2} diffractometer, interfaced to a *INDY* (*Silicon Graphics*) *UNIX* computer (Mo tube, 50 kV, 40 mA, monochromated Mo-K radiation, $\Delta\omega = 0.80 + 0.34$ tg θ).

Unit cell parameters[3] and orientation matrix were determined from a least-squares treatment of the *SET4*[4] setting angles of 22 reflections in the range 14.85° < θ < 18.69°. The unit cell was identified as monoclinic; reduced cell calculations did not indicate any higher metric lattice symmetry.[5] The space group $P2_1/n$ was derived from the systematic extinctions. Examination of the final atomic coordinates of the structure did not yield extra metric symmetry elements.[6,7]

The intensities of three standard reflections, monitored every three hours of X-ray exposure time, showed no greater fluctuations during data collection than those expected from Poisson statistics. Intensity data were corrected for Lorentz and polarization effects, scale variation, but not for absorption and reduced to F_o^2.[8]

The structure was solved by Patterson methods and extension of the model was accomplished by direct methods applied to difference structure factors using the program *DIRDIF*.[9] The positional and anisotropic thermal displacement parameters for the non-hydrogen atoms were refined. The hydrogen atoms were included in the final refinement riding on their carrier atoms with their positions calculated by using sp^2 or sp^3 hybridization at the C-atom as appropriate with $U_{iso} = c \times U_{equiv}$ of their parent atom, where c = 1.2 for the aromatic / non-methyl hydrogen atoms and c = 1.5 for the methyl hydrogen atoms and where values U_{equiv} are related to the atoms to which the H atoms are bonded. The methyl-groups were refined as rigid groups, which were allowed to rotate free.

Final refinement on F^2 carried out by full-matrix least-squares techniques converged at $wR(F^2)$ = 0.1998 for 15914 reflections with $F_o^2 \geq 0$ and $R(F)$ = 0.0840 for 7483 reflections with $F_o \geq 4.0\ \sigma(F_o)$ and 957 parameters.

The final difference Fourier map was essentially featureless: no significant peaks having chemical meaning above the general background were observed.

The positional and anisotropic thermal displacement parameters for the non-hydrogen atoms and isotropic thermal displacement parameters for hydrogen atoms were refined on F^2 with full-matrix least-squares procedures minimizing the function $Q = \Sigma_h [w(|F_o^2 - kF_c^2|)^2]$, where $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$, $P = [\max(F_o^2,0) + 2F_c^2] / 3$, F_o and F_c are the observed and calculated structure factor amplitudes, respectively; a and b were refined. Reflections were stated observed if satisfying $F^2 > 0$ criterion of observability.

Neutral atom scattering factors and anomalous dispersion corrections were taken from *International Tables of Crystallography.*[10] All calculations performed on the HP9000/735 computer at the University of Groningen with the program packages *SHELXL*[11] (least-square refinements), *PLATON*[12] (calculation of geometric data and the *ORTEP* illustrations) and a locally modified version of the program *PLUTO*[13] (preparation of illustrations).

Each asymmetric unit contains two formula unit molecules with no atom setting at special position. The monoclinic unit cell contains eight discrete units of each type of the title compound separated by normal van der Waals distances[16] (Fig. 2). No missed symmetry (*MISSYM*) 154.1 Å^3 voids in the unit cell were detected by procedures implemented in *PLATON.*[17,18]

Table 1.

a. Crystal data and details of the structure determination.

Moiety_ Formula	$C_{42}H_{62}LiN_6Si_2Y$
Formula_Weight, g.mol^{-1}	803.01
Crystal system	monoclinic
Space group, no.[18]	$P2_1/n$, 14
a, Å	19.695(3)
b, Å	19.968(3)
c, Å	23.502(3)
β, deg	101.37(1)
V, Å^3	9061(2)
Formula_*Z*	8
SpaceGroup_*Z*	4
Z' (= Formula_Z / SpaceGroup-*Z*)	2
ρ_{calc}, g.cm^{-3}	1.177
F(000), electrons	3408
μ(Mo K), cm^{-1}	13.71
color, habit	light yellow, parallelepiped
Approx. crystal dimension, mm	0.15 x 0.19 x 0.28

b. Data collection.

Radiation	Mo K
Wavelength, Å	0.71073
Monochromator	Graphite
Temperature, K	130
θ range; min. max., deg	1.02, 25.0
ω/2θ scan, deg	$\Delta\omega = 0.80 + 0.34 \text{ tg } \theta$
Index ranges	h: 0→23; k: 0→23; l: -27→27
Crystal-to-receiving-aperture-distance, mm	173
Horizontal-, vertical-aperture, mm	3.2 + tg θ; 4.0
Reference reflections,	3-33, 1.3
r.m.s. dev. in %	33-3, 0.8
	-333, 1.1
Drift correction	0.992 - 1.007
X-ray exposure time, h	202.9
Total data	17183
Unique data	15918
Data with criterion: ($F_o \geq 4.0\ \sigma\ (F_o)$)	7483

c. Refinement.

Number of reflections ($F_o^2 \geq 0$)	15914
Number of refined parameters	957
Final agreement factors:	
$wR(F^2) = [\Sigma[w(F_o^2 - F_c^2)^2] / \Sigma[w(F_o^2)^2]]^{1/2}$	0.1998
for $F_o^2 > 0$	
Weighting scheme: *a, b*	0.0907, 0.0
$w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$	
and $P = [\max(F_o^2,0) + 2F_c^2] / 3$	
$R(F) = \Sigma(\|\|F_o\| - \|F_c\|\|) / \Sigma\|F_o\|$	0.0840
for $F_o > 4.0\ \sigma\ (F_o)$	
GooF = S = $[\Sigma[w(F_o^2 - F_c^2)^2] / (n-p)]^{1/2}$	0.949
n = number of reflections	
p = number of parameters refined	
Residual electron density in final	
difference Fourier map, e/Å^3	-1.34, 0.82(11)
Max. (shift/σ) final cycle	< 0.001

Table 2a. Final fractional atomic coordinates and equivalent isotropic thermal displacement parameters for non-H atoms with s.u.'s in parentheses.

Atoms of the Asymmetric Unit.

Residue: 1.

Atom	*x*	*y*	*z*	U_{eq} (Å^2)*
Y(1)	0.63129(4)	0.08756(3)	0.26050(3)	0.0239(2)
Si(1)	0.59050(11)	0.12072(11)	0.41244(9)	0.0320(8)
Si(2)	0.67213(12)	0.02665(11)	0.11273(9)	0.0319(8)
N(1)	0.6436(3)	0.0994(3)	0.3650(2)	0.027(2)
N(2)	0.7407(3)	0.0751(3)	0.3303(3)	0.0257(19)
N(3)	0.8279(3)	-0.0271(3)	0.2981(3)	0.034(2)
N(4)	0.6559(3)	0.0185(3)	0.1833(3)	0.026(2)
N(5)	0.6336(3)	-0.0364(3)	0.2613(3)	0.0250(19)
N(6)	0.7013(3)	-0.0993(3)	0.3665(3)	0.035(2)
C(1)	0.6759(4)	0.1984(4)	0.2361(3)	0.038(3)
C(2)	0.6186(4)	0.2369(4)	0.2009(3)	0.034(3)
C(3)	0.5704(4)	0.2732(4)	0.2252(4)	0.039(3)
C(4)	0.5149(5)	0.3059(4)	0.1911(5)	0.055(4)
C(5)	0.5048(5)	0.3042(5)	0.1320(5)	0.052(4)
C(6)	0.5510(5)	0.2682(5)	0.1067(4)	0.051(3)
C(7)	0.6061(4)	0.2364(4)	0.1410(3)	0.035(3)
C(8)	0.5048(3)	0.1017(4)	0.2462(3)	0.036(3)
C(9)	0.4604(4)	0.0753(4)	0.1940(3)	0.027(3)
C(10)	0.4553(4)	0.1056(4)	0.1388(3)	0.040(3)
C(11)	0.4142(5)	0.0798(5)	0.0910(4)	0.056(4)
C(12)	0.3725(5)	0.0247(5)	0.0945(5)	0.058(4)
C(13)	0.3765(4)	-0.0060(5)	0.1459(4)	0.046(3)
C(14)	0.4195(4)	0.0185(4)	0.1955(4)	0.039(3)
C(15)	0.5377(4)	0.1954(4)	0.3834(3)	0.045(3)
C(16)	0.6330(4)	0.1390(4)	0.4886(3)	0.041(3)
C(17)	0.5329(4)	0.0479(4)	0.4181(3)	0.043(3)
C(18)	0.7129(4)	0.1052(3)	0.3709(3)	0.025(3)
C(19)	0.7556(4)	0.1451(4)	0.4185(3)	0.029(3)
C(20)	0.7982(4)	0.1133(4)	0.4661(3)	0.040(3)
C(21)	0.8339(5)	0.1511(5)	0.5103(4)	0.049(3)
C(22)	0.8312(5)	0.2195(5)	0.5086(4)	0.051(3)
C(23)	0.7919(5)	0.2519(4)	0.4624(4)	0.043(3)
C(24)	0.7543(4)	0.2152(4)	0.4180(4)	0.040(3)
C(25)	0.8144(4)	0.0905(4)	0.3278(3)	0.036(3)
C(26)	0.8357(4)	0.0439(3)	0.2836(3)	0.034(3)
C(27)	0.8796(4)	-0.0466(4)	0.3466(4)	0.047(3)
C(28)	0.8338(5)	-0.0684(4)	0.2470(4)	0.051(4)
C(29)	0.5983(5)	0.0630(4)	0.0606(3)	0.047(3)
C(30)	0.7501(4)	0.0834(4)	0.1216(4)	0.046(3)
C(31)	0.6975(5)	-0.0531(4)	0.0811(3)	0.049(3)
C(32)	0.6300(4)	-0.0353(4)	0.2062(3)	0.023(3)
C(33)	0.5933(4)	-0.0904(3)	0.1661(3)	0.026(3)
C(34)	0.5330(4)	-0.0775(4)	0.1307(3)	0.040(3)
C(35)	0.4980(5)	-0.1278(5)	0.0942(4)	0.049(3)
C(36)	0.5276(5)	-0.1913(5)	0.0959(4)	0.052(4)
C(37)	0.5907(5)	-0.2035(4)	0.1320(4)	0.045(3)

C(40)	0.6256(4)	-0.0879(4)	0.3540(3)	0.032(3)
C(41)	0.7286(5)	-0.0859(5)	0.4279(3)	0.059(4)
C(42)	0.7184(5)	-0.1675(4)	0.3512(4)	0.057(4)
Li(1)	0.7293(6)	-0.0283(6)	0.3128(5)	0.027(4)

Residue: 2.

Atom	*x*	*y*	*z*	U_{eq} (Å^2)*
Y(2)	0.13312(4)	0.10476(3)	0.25579(3)	0.0222(2)
Si(3)	0.29014(11)	0.09907(11)	0.38303(9)	0.0302(7)
Si(4)	-0.02416(12)	0.13366(12)	0.13301(11)	0.0372(8)
N(7)	0.2244(3)	0.1375(3)	0.3356(2)	0.0195(17)
N(8)	0.1511(3)	0.2193(3)	0.2933(3)	0.025(2)
N(9)	0.1311(3)	0.3203(3)	0.2071(3)	0.032(2)
N(10)	0.0636(3)	0.1394(3)	0.1653(3)	0.028(2)
N(11)	0.1790(3)	0.1507(3)	0.1746(2)	0.024(2)
N(12)	0.2943(3)	0.2370(3)	0.2133(3)	0.043(3)
C(43)	0.0316(4)	0.0733(4)	0.2949(4)	0.045(3)
C(44)	0.0307(3)	0.0749(4)	0.3572(3)	0.026(3)
C(45)	0.0651(4)	0.0281(4)	0.3967(4)	0.047(4)
C(46)	0.0653(4)	0.0313(5)	0.4546(4)	0.049(3)
C(47)	0.0311(5)	0.0809(5)	0.4778(3)	0.045(3)
C(48)	-0.0042(5)	0.1278(4)	0.4405(4)	0.049(3)
C(49)	-0.0035(4)	0.1255(4)	0.3825(3)	0.033(3)
C(50)	0.1821(4)	-0.0079(4)	0.2515(4)	0.048(3)
C(51)	0.1451(4)	-0.0512(4)	0.2062(4)	0.037(3)
C(52)	0.1660(5)	-0.0633(4)	0.1538(4)	0.050(3)
C(53)	0.1272(6)	-0.1043(5)	0.1102(4)	0.060(4)
C(54)	0.0665(6)	-0.1320(5)	0.1171(5)	0.062(4)
C(55)	0.0457(5)	-0.1208(5)	0.1679(5)	0.065(4)
C(56)	0.0804(5)	-0.0816(4)	0.2096(4)	0.042(3)
C(57)	0.2559(4)	0.0308(4)	0.4249(3)	0.040(3)
C(58)	0.3486(4)	0.0594(4)	0.3393(4)	0.052(3)
C(59)	0.3435(4)	0.1581(4)	0.4349(3)	0.044(3)
C(60)	0.1907(4)	0.1942(4)	0.3416(3)	0.026(3)
C(61)	0.1936(4)	0.2264(3)	0.3987(3)	0.026(3)
C(62)	0.1634(4)	0.1957(4)	0.4393(3)	0.030(3)
C(63)	0.1652(4)	0.2250(4)	0.4939(3)	0.038(3)
C(64)	0.2008(5)	0.2851(5)	0.5062(4)	0.055(4)
C(65)	0.2306(5)	0.3164(5)	0.4658(4)	0.062(4)
C(66)	0.2275(4)	0.2881(4)	0.4121(3)	0.040(3)
C(67)	0.1022(4)	0.2746(4)	0.2970(3)	0.036(3)
C(68)	0.0771(5)	0.3030(4)	0.2401(4)	0.054(3)
C(69)	0.1709(5)	0.3779(5)	0.2323(5)	0.088(5)
C(70)	0.0981(6)	0.3351(5)	0.1471(4)	0.086(5)
C(71)	-0.0473(4)	0.1622(5)	0.0552(4)	0.062(4)
C(72)	-0.0536(4)	0.0465(4)	0.1402(4)	0.042(3)
C(73)	-0.0733(4)	0.1896(4)	0.1746(4)	0.053(3)
C(74)	0.1178(4)	0.1395(3)	0.1390(3)	0.028(3)
C(75)	0.1144(4)	0.1241(4)	0.0758(3)	0.028(3)
C(76)	0.1304(4)	0.1728(4)	0.0387(3)	0.035(3)
C(77)	0.1269(5)	0.1586(4)	-0.0183(4)	0.046(3)
C(78)	0.1104(5)	0.0940(5)	-0.0396(3)	0.050(4)
C(79)	0.0947(5)	0.0463(4)	-0.0029(4)	0.052(4)

C(82)	0.3017(5)	0.1648(5)	0.1982(4)	0.058(4)
C(83)	0.3433(5)	0.2499(5)	0.2672(4)	0.062(4)
C(84)	0.3090(5)	0.2816(5)	0.1695(4)	0.066(4)
Li(2)	0.1935(6)	0.2376(6)	0.2214(5)	0.026(4)

*) $U_{eq} = 1/3\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$ [20]

Table 2b: Hydrogen parameters

H(1)	0.69511(-)	0.22315(-)	0.2722(-)	0.04527(-)
H(1')	0.71342(-)	0.19199(-)	0.2141(-)	0.04527(-)
H(3)	0.57596(-)	0.27538(-)	0.26622(-)	0.04663(-)
H(4)	0.4833(-)	0.32984(-)	0.20918(-)	0.06626(-)
H(5)	0.46701(-)	0.32722(-)	0.10884(-)	0.06257(-)
H(6)	0.54466(-)	0.2655(-)	0.06567(-)	0.06094(-)
H(7)	0.63744(-)	0.2126(-)	0.12239(-)	0.04173(-)
H(8)	0.48971(-)	0.08111(-)	0.27995(-)	0.04392(-)
H(8')	0.49543(-)	0.15032(-)	0.24769(-)	0.04392(-)
H(10)	0.48144(-)	0.14484(-)	0.13527(-)	0.0481(-)
H(11)	0.41382(-)	0.09986(-)	0.05424(-)	0.06709(-)
H(12)	0.34157(-)	0.00874(-)	0.06105(-)	0.06902(-)
H(13)	0.34959(-)	-0.04498(-)	0.1484(-)	0.0548(-)
H(14)	0.42117(-)	-0.00393(-)	0.23143(-)	0.04695(-)
H(15)	0.5685(-)	0.23134(-)	0.37549(-)	0.06641(-)
H(15')	0.51125(-)	0.21088(-)	0.4121(-)	0.06641(-)
H(15'')	0.50578(-)	0.18326(-)	0.34743(-)	0.06641(-)
H(16)	0.66199(-)	0.10093(-)	0.50451(-)	0.06175(-)
H(16')	0.5975(-)	0.1466(-)	0.51187(-)	0.06175(-)
H(16'')	0.66181(-)	0.17915(-)	0.48963(-)	0.06175(-)
H(17)	0.50918(-)	0.03431(-)	0.37912(-)	0.06427(-)
H(17')	0.49849(-)	0.06056(-)	0.44108(-)	0.06427(-)
H(17'')	0.56071(-)	0.01041(-)	0.43695(-)	0.06427(-)
H(20)	0.80204(-)	0.06588(-)	0.46737(-)	0.04906(-)
H(21)	0.86119(-)	0.12953(-)	0.54301(-)	0.05861(-)
H(22)	0.85682(-)	0.24467(-)	0.53984(-)	0.06122(-)
H(23)	0.79081(-)	0.29945(-)	0.4611(-)	0.05172(-)
H(24)	0.72652(-)	0.23775(-)	0.3861(-)	0.0478(-)
H(25)	0.84451(-)	0.08358(-)	0.36635(-)	0.04272(-)
H(25')	0.8188(-)	0.13773(-)	0.31616(-)	0.04272(-)
H(26)	0.80699(-)	0.05333(-)	0.24489(-)	0.04007(-)
H(26')	0.88464(-)	0.05269(-)	0.28157(-)	0.04007(-)
H(27)	0.92555(-)	-0.04027(-)	0.33738(-)	0.07078(-)
H(27')	0.87323(-)	-0.09385(-)	0.3555(-)	0.07078(-)
H(27'')	0.87549(-)	-0.01907(-)	0.3803(-)	0.07078(-)
H(28)	0.87643(-)	-0.05669(-)	0.23377(-)	0.07643(-)
H(28')	0.79379(-)	-0.05996(-)	0.21571(-)	0.07643(-)
H(28'')	0.83494(-)	-0.11588(-)	0.25767(-)	0.07643(-)
H(29)	0.56032(-)	0.03049(-)	0.05285(-)	0.07027(-)
H(29')	0.61309(-)	0.07399(-)	0.02427(-)	0.07027(-)
H(29'')	0.58239(-)	0.10381(-)	0.07715(-)	0.07027(-)
H(30)	0.74343(-)	0.12169(-)	0.14601(-)	0.06888(-)
H(30')	0.7557(-)	0.09955(-)	0.08337(-)	0.06888(-)
H(30'')	0.79153(-)	0.05857(-)	0.13987(-)	0.06888(-)

H(31")	0.65712(-)	-0.08265(-)	0.07178(-)	0.0735(-)
H(34)	0.51338(-)	-0.03393(-)	0.1301(-)	0.04797(-)
H(35)	0.45503(-)	-0.11859(-)	0.06897(-)	0.05932(-)
H(36)	0.50435(-)	-0.22613(-)	0.0722(-)	0.06309(-)
H(37)	0.61176(-)	-0.24628(-)	0.13171(-)	0.05439(-)
H(38)	0.66559(-)	-0.16358(-)	0.19429(-)	0.04737(-)
H(39)	0.5975(-)	-0.13002(-)	0.27129(-)	0.03997(-)
H(39')	0.54533(-)	-0.07201(-)	0.28261(-)	0.03997(-)
H(40)	0.61564(-)	-0.04512(-)	0.37206(-)	0.03911(-)
H(40')	0.60283(-)	-0.12423(-)	0.37192(-)	0.03911(-)
H(41)	0.70795(-)	-0.11725(-)	0.45175(-)	0.08791(-)
H(41')	0.71694(-)	-0.03988(-)	0.4371(-)	0.08791(-)
H(41")	0.77898(-)	-0.09137(-)	0.43614(-)	0.08791(-)
H(42)	0.76812(-)	-0.17526(-)	0.36446(-)	0.08486(-)
H(42')	0.70618(-)	-0.17308(-)	0.30902(-)	0.08486(-)
H(42")	0.69227(-)	-0.19964(-)	0.36994(-)	0.08486(-)
H(43)	-0.00686(-)	0.10224(-)	0.27559(-)	0.05437(-)
H(43')	0.01948(-)	0.02698(-)	0.28163(-)	0.05437(-)
H(45)	0.0893(-)	-0.00743(-)	0.38261(-)	0.05655(-)
H(46)	0.08973(-)	-0.00179(-)	0.47957(-)	0.05848(-)
H(47)	0.03183(-)	0.08285(-)	0.51831(-)	0.05413(-)
H(48)	-0.02932(-)	0.16211(-)	0.45521(-)	0.05887(-)
H(49)	-0.02705(-)	0.15963(-)	0.35817(-)	0.03939(-)
H(50)	0.23043(-)	-0.00334(-)	0.24597(-)	0.0575(-)
H(50')	0.18365(-)	-0.03013(-)	0.28941(-)	0.0575(-)
H(52)	0.20754(-)	-0.04339(-)	0.1472(-)	0.05976(-)
H(53)	0.14392(-)	-0.11267(-)	0.07562(-)	0.07193(-)
H(54)	0.03945(-)	-0.15827(-)	0.08727(-)	0.07383(-)
H(55)	0.00444(-)	-0.14182(-)	0.17402(-)	0.07806(-)
H(56)	0.06126(-)	-0.07356(-)	0.24304(-)	0.05098(-)
H(57)	0.23083(-)	0.05079(-)	0.45277(-)	0.06065(-)
H(57')	0.29464(-)	0.00399(-)	0.44577(-)	0.06065(-)
H(57")	0.22447(-)	0.00196(-)	0.39798(-)	0.06065(-)
H(58)	0.32159(-)	0.02972(-)	0.31019(-)	0.07735(-)
H(58')	0.38432(-)	0.03342(-)	0.36495(-)	0.07735(-)
H(58")	0.3706(-)	0.09432(-)	0.31988(-)	0.07735(-)
H(59)	0.36418(-)	0.19186(-)	0.41316(-)	0.0665(-)
H(59')	0.38037(-)	0.13327(-)	0.46035(-)	0.0665(-)
H(59")	0.31414(-)	0.18031(-)	0.45829(-)	0.0665(-)
H(62)	0.14078(-)	0.15386(-)	0.43052(-)	0.0357(-)
H(63)	0.14281(-)	0.20441(-)	0.52166(-)	0.04535(-)
H(64)	0.20433(-)	0.30474(-)	0.54345(-)	0.06508(-)
H(65)	0.25364(-)	0.35793(-)	0.47484(-)	0.07479(-)
H(66)	0.24827(-)	0.31009(-)	0.384(-)	0.04827(-)
H(67)	0.06267(-)	0.25757(-)	0.31308(-)	0.04332(-)
H(67')	0.12578(-)	0.30975(-)	0.32349(-)	0.04332(-)
H(68)	0.05034(-)	0.34394(-)	0.24462(-)	0.06461(-)
H(68')	0.04486(-)	0.2706(-)	0.21704(-)	0.06461(-)
H(69)	0.20345(-)	0.39088(-)	0.20763(-)	0.13227(-)
H(69')	0.19663(-)	0.36641(-)	0.2711(-)	0.13227(-)
H(69")	0.13946(-)	0.41526(-)	0.23505(-)	0.13227(-)
H(70)	0.07174(-)	0.29595(-)	0.12996(-)	0.12902(-)
H(70')	0.13369(-)	0.34596(-)	0.1247(-)	0.12902(-)
H(70")	0.06672(-)	0.37335(-)	0.14631(-)	0.12902(-)
H(71)	-0.02474(-)	0.20521(-)	0.05112(-)	0.0936(-)

H(72)	-0.01925(-)	0.01526(-)	0.13032(-)	0.06288(-)
H(72')	-0.09815(-)	0.03946(-)	0.11382(-)	0.06288(-)
H(72")	-0.05882(-)	0.03853(-)	0.18025(-)	0.06288(-)
H(73)	-0.05322(-)	0.18658(-)	0.21611(-)	0.07966(-)
H(73')	-0.12193(-)	0.17556(-)	0.16784(-)	0.07966(-)
H(73")	-0.07053(-)	0.23597(-)	0.16154(-)	0.07966(-)
H(76)	0.14376(-)	0.21623(-)	0.05312(-)	0.04186(-)
H(77)	0.13575(-)	0.19281(-)	-0.04391(-)	0.05594(-)
H(78)	0.11019(-)	0.08365(-)	-0.07911(-)	0.06009(-)
H(79)	0.08231(-)	0.00261(-)	-0.01729(-)	0.06186(-)
H(80)	0.08532(-)	0.02691(-)	0.08028(-)	0.04003(-)
H(81)	0.25104(-)	0.09149(-)	0.14764(-)	0.04703(-)
H(81')	0.24334(-)	0.16381(-)	0.11723(-)	0.04703(-)
H(82)	0.34545(-)	0.15902(-)	0.18406(-)	0.06929(-)
H(82')	0.30496(-)	0.13745(-)	0.23381(-)	0.06929(-)
H(83)	0.39001(-)	0.23756(-)	0.26243(-)	0.0932(-)
H(83')	0.3306(-)	0.22323(-)	0.29851(-)	0.0932(-)
H(83")	0.34229(-)	0.29758(-)	0.2769(-)	0.0932(-)
H(84)	0.30961(-)	0.32783(-)	0.18357(-)	0.09919(-)
H(84')	0.27311(-)	0.27694(-)	0.13432(-)	0.09919(-)
H(84")	0.35421(-)	0.27047(-)	0.16063(-)	0.09919(-)

Table 3: Anisotropic (displacement) parameters (Å^2)

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Y(1)	0.0200(4)	0.0203(4)	0.0304(4)	-0.0005(4)	0.0027(3)	0.0001(4)
Si(1)	0.0289(13)	0.0350(14)	0.0320(13)	-0.0055(10)	0.0059(10)	0.0049(10)
Si(2)	0.0349(13)	0.0310(13)	0.0329(13)	0.0074(11)	0.0147(11)	0.0085(11)
N(1)	0.029(4)	0.022(4)	0.029(4)	-0.001(3)	0.001(3)	0.006(3)
N(2)	0.014(3)	0.020(3)	0.046(4)	-0.006(3)	0.013(3)	-0.002(3)
N(3)	0.032(4)	0.027(4)	0.046(4)	0.003(3)	0.016(3)	0.004(3)
N(4)	0.017(3)	0.027(4)	0.037(4)	-0.003(3)	0.009(3)	0.000(3)
N(5)	0.024(3)	0.018(3)	0.032(4)	0.003(3)	0.003(3)	-0.010(3)
N(6)	0.049(4)	0.025(4)	0.030(4)	0.007(3)	0.007(3)	-0.003(3)
C(1)	0.040(5)	0.038(5)	0.034(5)	0.008(4)	0.006(4)	-0.004(4)
C(2)	0.041(5)	0.023(5)	0.041(5)	0.000(4)	0.016(4)	-0.019(4)
C(3)	0.049(6)	0.028(5)	0.044(5)	-0.009(4)	0.021(5)	-0.006(4)
C(4)	0.040(6)	0.026(5)	0.108(9)	-0.006(6)	0.033(6)	-0.003(4)
C(5)	0.047(6)	0.040(6)	0.063(7)	0.010(5)	-0.003(5)	-0.007(5)
C(6)	0.055(6)	0.050(6)	0.048(6)	0.002(5)	0.010(5)	-0.019(5)
C(7)	0.038(5)	0.036(5)	0.033(5)	0.012(4)	0.014(4)	0.001(4)
C(8)	0.024(4)	0.026(5)	0.064(6)	-0.002(4)	0.021(4)	-0.005(4)
C(9)	0.020(4)	0.022(4)	0.036(5)	-0.006(4)	0.001(4)	0.005(3)
C(10)	0.043(5)	0.037(5)	0.045(6)	0.008(5)	0.021(5)	0.013(4)
C(11)	0.045(6)	0.082(8)	0.040(6)	0.010(6)	0.004(5)	0.022(6)
C(12)	0.030(5)	0.072(8)	0.067(7)	-0.033(6)	0.001(5)	0.006(5)
C(13)	0.031(5)	0.051(6)	0.050(6)	-0.018(5)	-0.003(5)	-0.015(4)
C(14)	0.030(5)	0.031(5)	0.058(6)	-0.001(4)	0.015(4)	0.008(4)
C(15)	0.034(5)	0.047(6)	0.052(6)	-0.006(5)	0.007(4)	0.017(4)
C(16)	0.047(6)	0.051(6)	0.024(5)	0.001(4)	0.003(4)	0.009(5)
C(17)	0.028(5)	0.059(6)	0.043(5)	-0.005(5)	0.010(4)	-0.002(4)
C(18)	0.027(4)	0.012(4)	0.035(5)	0.003(3)	0.004(4)	0.000(3)
C(19)	0.033(5)	0.026(5)	0.029(5)	-0.006(4)	0.007(4)	-0.006(4)
C(20)	0.043(5)	0.027(5)	0.047(5)	-0.012(4)	-0.002(4)	-0.003(4)
C(21)	0.047(6)	0.050(6)	0.041(6)	-0.001(5)	-0.012(5)	0.003(5)
C(22)	0.050(6)	0.050(6)	0.050(6)	-0.018(5)	0.001(5)	-0.020(5)
C(23)	0.064(6)	0.024(5)	0.044(6)	-0.016(4)	0.016(5)	-0.018(5)
C(24)	0.052(6)	0.023(5)	0.045(5)	0.004(4)	0.011(5)	-0.007(4)
C(25)	0.022(4)	0.024(5)	0.062(6)	-0.006(4)	0.010(4)	-0.007(4)
C(26)	0.023(4)	0.026(5)	0.059(6)	0.002(4)	0.025(4)	0.003(4)
C(27)	0.027(5)	0.044(6)	0.063(6)	0.008(5)	-0.009(5)	0.003(4)
C(28)	0.046(6)	0.041(6)	0.069(7)	-0.001(5)	0.018(5)	0.005(5)
C(29)	0.067(7)	0.035(5)	0.045(6)	0.006(4)	0.027(5)	0.002(5)
C(30)	0.039(5)	0.049(6)	0.056(6)	0.020(5)	0.025(5)	0.002(5)
C(31)	0.066(7)	0.054(6)	0.030(5)	0.002(4)	0.015(5)	0.020(5)
C(32)	0.018(4)	0.025(4)	0.028(5)	0.002(4)	0.007(3)	0.006(3)
C(33)	0.035(5)	0.019(4)	0.025(4)	-0.007(3)	0.009(4)	-0.003(4)
C(34)	0.040(5)	0.030(5)	0.043(5)	0.000(4)	-0.010(4)	0.011(4)
C(35)	0.053(6)	0.046(6)	0.040(6)	-0.013(5)	-0.012(5)	-0.003(5)
C(36)	0.067(7)	0.036(6)	0.055(6)	-0.023(5)	0.014(5)	-0.022(5)
C(37)	0.055(6)	0.030(5)	0.049(6)	-0.014(4)	0.007(5)	0.008(5)
C(38)	0.038(5)	0.029(5)	0.053(6)	0.005(4)	0.012(4)	0.005(4)
C(39)	0.030(5)	0.029(5)	0.045(5)	-0.003(4)	0.015(4)	-0.012(4)
C(40)	0.037(5)	0.038(5)	0.021(4)	0.002(4)	0.003(4)	-0.006(4)
C(41)	0.069(7)	0.072(7)	0.026(5)	0.015(5)	-0.014(5)	-0.024(6)

Y(2)	0.0198(4)	0.0170(4)	0.0294(4)	-0.0021(3)	0.0037(3)	-0.0010(3)
Si(3)	0.0247(12)	0.0311(13)	0.0338(13)	-0.0019(11)	0.0033(10)	0.0018(11)
Si(4)	0.0228(12)	0.0322(13)	0.0508(16)	-0.0019(12)	-0.0068(11)	0.0010(11)
N(7)	0.022(3)	0.018(3)	0.018(3)	0.000(3)	0.003(3)	0.004(3)
N(8)	0.031(4)	0.016(3)	0.029(4)	-0.002(3)	0.012(3)	-0.004(3)
N(9)	0.041(4)	0.019(4)	0.037(4)	0.005(3)	0.013(3)	0.006(3)
N(10)	0.024(4)	0.020(3)	0.040(4)	-0.001(3)	0.009(3)	-0.002(3)
N(11)	0.026(4)	0.018(3)	0.029(4)	-0.004(3)	0.009(3)	0.003(3)
N(12)	0.036(4)	0.021(4)	0.075(6)	-0.019(4)	0.018(4)	-0.010(3)
C(43)	0.034(5)	0.034(5)	0.074(7)	-0.007(5)	0.027(5)	-0.010(4)
C(44)	0.015(4)	0.023(4)	0.038(5)	0.004(4)	0.000(4)	-0.008(3)
C(45)	0.039(5)	0.024(5)	0.085(8)	0.003(5)	0.027(5)	0.000(4)
C(46)	0.036(5)	0.048(6)	0.056(6)	0.027(5)	-0.008(5)	-0.011(5)
C(47)	0.050(6)	0.056(6)	0.027(5)	0.012(5)	0.002(4)	-0.015(5)
C(48)	0.055(6)	0.034(5)	0.068(7)	0.004(5)	0.036(5)	-0.006(5)
C(49)	0.018(4)	0.035(5)	0.044(5)	0.010(4)	0.003(4)	0.003(4)
C(50)	0.040(5)	0.022(5)	0.070(7)	-0.004(5)	-0.016(5)	0.008(4)
C(51)	0.041(5)	0.010(4)	0.057(6)	-0.005(4)	-0.001(5)	0.014(4)
C(52)	0.054(6)	0.022(5)	0.069(7)	0.003(5)	0.000(5)	0.015(4)
C(53)	0.087(8)	0.040(6)	0.054(6)	0.007(5)	0.017(6)	0.032(6)
C(54)	0.069(8)	0.045(6)	0.062(7)	-0.008(6)	-0.009(6)	0.001(6)
C(55)	0.049(7)	0.037(6)	0.098(9)	-0.005(6)	-0.015(6)	0.002(5)
C(56)	0.059(6)	0.021(5)	0.040(5)	-0.001(4)	-0.008(5)	-0.006(5)
C(57)	0.039(5)	0.038(5)	0.041(5)	0.003(4)	0.000(4)	0.000(4)
C(58)	0.036(5)	0.041(6)	0.081(7)	-0.005(5)	0.020(5)	0.014(4)
C(59)	0.033(5)	0.051(6)	0.042(5)	0.001(5)	-0.009(4)	0.002(4)
C(60)	0.028(4)	0.028(5)	0.027(5)	-0.002(4)	0.016(4)	-0.009(4)
C(61)	0.020(4)	0.021(4)	0.035(5)	-0.004(4)	0.002(4)	-0.003(3)
C(62)	0.031(5)	0.026(4)	0.032(5)	0.002(4)	0.006(4)	0.002(4)
C(63)	0.045(5)	0.042(5)	0.031(5)	-0.002(4)	0.017(4)	-0.004(4)
C(64)	0.086(8)	0.051(6)	0.032(5)	-0.011(5)	0.024(5)	0.002(6)
C(65)	0.104(9)	0.039(6)	0.052(6)	-0.018(5)	0.035(6)	-0.026(6)
C(66)	0.053(6)	0.034(5)	0.040(5)	-0.008(4)	0.024(5)	-0.014(4)
C(67)	0.042(5)	0.028(5)	0.042(5)	0.011(4)	0.016(4)	0.011(4)
C(68)	0.045(6)	0.035(5)	0.083(7)	0.012(5)	0.015(5)	0.018(5)
C(69)	0.073(8)	0.029(6)	0.176(13)	-0.029(7)	0.059(8)	-0.025(6)
C(70)	0.123(11)	0.073(8)	0.054(7)	0.013(6)	-0.002(7)	0.046(8)
C(71)	0.029(5)	0.067(7)	0.080(7)	0.017(6)	-0.017(5)	0.005(5)
C(72)	0.022(5)	0.040(5)	0.057(6)	-0.011(5)	-0.009(4)	-0.009(4)
C(73)	0.031(5)	0.040(6)	0.081(7)	-0.012(5)	-0.007(5)	0.009(4)
C(74)	0.038(5)	0.007(4)	0.038(5)	0.004(3)	0.005(4)	0.007(4)
C(75)	0.036(5)	0.023(4)	0.023(4)	0.005(4)	0.002(4)	0.007(4)
C(76)	0.041(5)	0.035(5)	0.025(5)	0.005(4)	-0.002(4)	-0.003(4)
C(77)	0.063(6)	0.043(6)	0.034(5)	0.009(4)	0.012(5)	-0.017(5)
C(78)	0.073(7)	0.058(7)	0.020(5)	-0.006(5)	0.010(4)	-0.004(6)
C(79)	0.084(8)	0.032(5)	0.044(6)	-0.009(5)	0.026(5)	-0.004(5)
C(80)	0.039(5)	0.031(5)	0.024(5)	0.000(4)	-0.008(4)	-0.008(4)
C(81)	0.040(5)	0.034(5)	0.046(5)	-0.013(4)	0.016(4)	0.001(4)
C(82)	0.048(6)	0.055(7)	0.073(7)	-0.006(5)	0.020(6)	0.006(5)
C(83)	0.035(6)	0.063(7)	0.084(8)	0.016(6)	0.002(5)	-0.010(5)
C(84)	0.047(6)	0.098(9)	0.057(7)	0.025(6)	0.017(5)	0.010(6)
Li(2)	0.021(7)	0.024(7)	0.037(8)	0.005(6)	0.012(6)	-0.002(6)

Table 4. Selected data on the geometry.
Standard deviations in the last decimal place are given in parentheses.

Residue 1.

Interatomic Distances (Å)

Y(1)	-N(1)	2.431(5)
Y(1)	-N(2)	2.450(7)
Y(1)	-N(4)	2.403(7)
Y(1)	-N(5)	2.476(6)
Y(1)	-C(1)	2.490(8)
Y(1)	-C(8)	2.463(6)
Y(1)	-C(18)	2.792(7)
Y(1)	-C(32)	2.763(8)
Si(1)	-N(1)	1.726(6)
Si(1)	-C(15)	1.868(8)
Si(1)	-C(16)	1.857(7)
Si(1)	-C(17)	1.865(8)
Si(2)	-N(4)	1.757(7)
Si(2)	-C(29)	1.855(9)
Si(2)	-C(30)	1.887(8)
Si(2)	-C(31)	1.867(8)
N(1)	-C(18)	1.349(10)
N(2)	-C(18)	1.333(10)
N(2)	-C(25)	1.496(10)
N(2)	-Li(1)	2.109(13)
N(3)	-C(26)	1.473(9)
N(3)	-C(27)	1.425(11)
N(3)	-C(28)	1.480(11)
N(3)	-Li(1)	2.039(14)
N(4)	-C(32)	1.346(10)
N(5)	-C(32)	1.283(10)
N(5)	-C(39)	1.467(10)
N(5)	-Li(1)	2.036(14)
N(6)	-C(40)	1.479(10)
N(6)	-C(41)	1.462(10)
N(6)	-C(42)	1.465(10)
N(6)	-Li(1)	2.045(13)
C(1)	-C(2)	1.477(11)
C(2)	-C(3)	1.402(11)
C(2)	-C(7)	1.381(10)
C(3)	-C(4)	1.385(13)
C(4)	-C(5)	1.364(16)
C(5)	-C(6)	1.383(14)
C(6)	-C(7)	1.373(12)
C(8)	-C(9)	1.458(10)
C(9)	-C(10)	1.417(10)
C(9)	-C(14)	1.396(11)
C(10)	-C(11)	1.351(12)
C(11)	-C(12)	1.385(14)
C(12)	-C(13)	1.343(15)
C(13)	-C(14)	1.388(13)
C(18)	-C(19)	1.491(10)
C(19)	-C(20)	1.41(1)
C(19)	-C(24)	1.400(11)
C(20)	-C(21)	1.362(12)
C(21)	-C(22)	1.367(14)
C(22)	-C(23)	1.366(13)
C(23)	-C(24)	1.367(13)
C(25)	-C(26)	1.514(10)
C(32)	-C(33)	1.534(10)
C(33)	-C(34)	1.334(11)
C(33)	-C(38)	1.405(10)
C(34)	-C(35)	1.409(12)
C(35)	-C(36)	1.393(14)
C(36)	-C(37)	1.381(14)
C(37)	-C(38)	1.372(12)
C(39)	-C(40)	1.534(10)

Bond angles (deg.)

N(1)	-Y(1)	-N(2)	55.5(2)
N(1)	-Y(1)	-N(4)	145.4(2)
N(1)	-Y(1)	-N(5)	95.3(2)
N(1)	-Y(1)	-C(1)	100.2(2)
N(1)	-Y(1)	-C(8)	91.3(2)
N(1)	-Y(1)	-C(18)	28.9(2)
N(1)	-Y(1)	-C(32)	122.9(2)
N(2)	-Y(1)	-N(4)	99.5(2)
N(2)	-Y(1)	-N(5)	83.2(2)
N(2)	-Y(1)	-C(1)	86.6(2)
N(2)	-Y(1)	-C(8)	146.6(2)
N(2)	-Y(1)	-C(18)	28.5(2)
N(2)	-Y(1)	-C(32)	98.4(2)
C(26)	-N(3)	-C(27)	110.9(6)
C(26)	-N(3)	-C(28)	108.7(6)
C(26)	-N(3)	-Li(1)	101.3(6)
C(27)	-N(3)	-C(28)	109.8(6)
C(27)	-N(3)	-Li(1)	114.6(6)
C(28)	-N(3)	-Li(1)	111.1(6)
Y(1)	-N(4)	-Si(2)	139.5(3)
Y(1)	-N(4)	-C(32)	90.4(5)
Si(2)	-N(4)	-C(32)	127.2(5)
Y(1)	-N(5)	-C(32)	88.7(5)
Y(1)	-N(5)	-C(39)	131.2(5)
Y(1)	-N(5)	-Li(1)	86.5(4)
C(32)	-N(5)	-C(39)	121.7(6)

N(4)	-Y(1)	-C(8)	108.2(2)
N(4)	-Y(1)	-C(18)	127.5(2)
N(4)	-Y(1)	-C(32)	29.2(2)
N(5)	-Y(1)	-C(1)	152.1(2)
N(5)	-Y(1)	-C(8)	97.6(2)
N(5)	-Y(1)	-C(18)	96.5(2)
N(5)	-Y(1)	-C(32)	27.7(2)
C(1)	-Y(1)	-C(8)	105.1(3)
C(1)	-Y(1)	-C(18)	86.4(2)
C(1)	-Y(1)	-C(32)	130.9(2)
C(8)	-Y(1)	-C(18)	119.6(2)
C(8)	-Y(1)	-C(32)	97.0(2)
C(18)	-Y(1)	-C(32)	119.5(2)
N(1)	-Si(1)	-C(15)	109.0(3)
N(1)	-Si(1)	-C(16)	117.1(3)
N(1)	-Si(1)	-C(17)	108.0(3)
C(15)	-Si(1)	-C(16)	107.9(3)
C(15)	-Si(1)	-C(17)	110.2(4)
C(16)	-Si(1)	-C(17)	104.4(3)
N(4)	-Si(2)	-C(29)	113.3(4)
N(4)	-Si(2)	-C(30)	104.3(4)
N(4)	-Si(2)	-C(31)	114.2(3)
C(29)	-Si(2)	-C(30)	110.7(4)
C(29)	-Si(2)	-C(31)	107.9(4)
C(30)	-Si(2)	-C(31)	106.3(4)
Y(1)	-N(1)	-Si(1)	136.4(3)
Y(1)	-N(1)	-C(18)	90.5(4)
Si(1)	-N(1)	-C(18)	129.9(5)
Y(1)	-N(2)	-C(18)	90.1(4)
Y(1)	-N(2)	-C(25)	133.2(5)
Y(1)	-N(2)	-Li(1)	85.6(4)
C(18)	-N(2)	-C(25)	118.3(6)
C(18)	-N(2)	-Li(1)	122.6(6)
C(25)	-N(2)	-Li(1)	105.1(5)
C(20)	-C(21)	-C(22)	121.3(9)
C(21)	-C(22)	-C(23)	120.6(9)
C(22)	-C(23)	-C(24)	119.3(8)
C(19)	-C(24)	-C(23)	121.5(8)
N(2)	-C(25)	-C(26)	107.6(6)
N(3)	-C(26)	-C(25)	112.2(6)
Y(1)	-C(32)	-N(4)	60.4(4)
Y(1)	-C(32)	-N(5)	63.6(4)
Y(1)	-C(32)	-C(33)	151.8(5)
N(4)	-C(32)	-N(5)	117.9(7)
N(4)	-C(32)	-C(33)	119.8(6)
N(5)	-C(32)	-C(33)	122.2(7)
C(32)	-C(33)	-C(34)	119.9(6)
C(32)	-C(33)	-C(38)	119.1(7)
C(40)	-N(6)	-C(41)	109.1(6)
C(40)	-N(6)	-C(42)	111.5(6)
C(40)	-N(6)	-Li(1)	98.9(6)
C(41)	-N(6)	-C(42)	110.8(7)
C(41)	-N(6)	-Li(1)	113.0(6)
C(42)	-N(6)	-Li(1)	113.0(6)
Y(1)	-C(1)	-C(2)	109.1(5)
C(1)	-C(2)	-C(3)	123.0(7)
C(1)	-C(2)	-C(7)	122.0(7)
C(3)	-C(2)	-C(7)	114.8(7)
C(2)	-C(3)	-C(4)	121.9(9)
C(3)	-C(4)	-C(5)	121.2(9)
C(4)	-C(5)	-C(6)	118.3(9)
C(5)	-C(6)	-C(7)	119.9(9)
C(2)	-C(7)	-C(6)	123.8(8)
Y(1)	-C(8)	-C(9)	119.7(5)
C(8)	-C(9)	-C(10)	122.6(7)
C(8)	-C(9)	-C(14)	121.7(7)
C(10)	-C(9)	-C(14)	115.7(7)
C(9)	-C(10)	-C(11)	121.4(8)
C(10)	-C(11)	-C(12)	121.2(9)
C(11)	-C(12)	-C(13)	119.1(10)
C(12)	-C(13)	-C(14)	120.8(9)
C(9)	-C(14)	-C(13)	121.7(8)
Y(1)	-C(18)	-N(1)	60.6(3)
Y(1)	-C(18)	-N(2)	61.3(4)
Y(1)	-C(18)	-C(19)	154.3(5)
N(1)	-C(18)	-N(2)	115.9(6)
N(1)	-C(18)	-C(19)	122.2(6)
N(2)	-C(18)	-C(19)	121.8(7)
C(18)	-C(19)	-C(20)	120.9(7)
C(18)	-C(19)	-C(24)	121.4(7)
C(20)	-C(19)	-C(24)	117.6(7)
C(19)	-C(20)	-C(21)	119.5(8)
C(34)	-C(33)	-C(38)	120.9(7)
C(33)	-C(34)	-C(35)	120.7(8)
C(34)	-C(35)	-C(36)	118.7(9)
C(35)	-C(36)	-C(37)	119.9(9)
C(36)	-C(37)	-C(38)	120.6(8)
C(33)	-C(38)	-C(37)	119.1(8)
N(5)	-C(39)	-C(40)	108.9(6)
N(6)	-C(40)	-C(39)	112.9(6)
N(2)	-Li(1)	-N(3)	87.3(5)
N(2)	-Li(1)	-N(5)	104.1(6)
N(2)	-Li(1)	-N(6)	126.1(6)
N(3)	-Li(1)	-N(5)	134.5(7)
N(3)	-Li(1)	-N(6)	119.6(6)
N(5)	-Li(1)	-N(6)	88.9(5)

Residue 2.

Interatomic Distances (Å)

Y(2)	-N(7)	2.418(5)
Y(2)	-N(8)	2.452(6)
N(12)	-Li(2)	2.031(14)
C(43)	-C(44)	1.468(11)

Y(2)	-C(43)	2.442(8)	C(45)	-C(46)	1.361(13)
Y(2)	-C(50)	2.458(8)	C(46)	-C(47)	1.370(13)
Y(2)	-C(60)	2.764(8)	C(47)	-C(48)	1.374(13)
Y(2)	-C(74)	2.789(7)	C(48)	-C(49)	1.367(12)
Si(3)	-N(7)	1.714(6)	C(50)	-C(51)	1.451(12)
Si(3)	-C(57)	1.882(8)	C(51)	-C(52)	1.394(13)
Si(3)	-C(58)	1.864(9)	C(51)	-C(56)	1.428(13)
Si(3)	-C(59)	1.864(8)	C(52)	-C(53)	1.413(13)
Si(4)	-N(10)	1.749(7)	C(53)	-C(54)	1.356(17)
Si(4)	-C(71)	1.884(10)	C(54)	-C(55)	1.355(16)
Si(4)	-C(72)	1.853(8)	C(55)	-C(56)	1.333(14)
Si(4)	-C(73)	1.874(9)	C(60)	-C(61)	1.479(10)
N(7)	-C(60)	1.334(10)	C(61)	-C(62)	1.365(10)
N(8)	-C(60)	1.341(10)	C(61)	-C(66)	1.407(10)
N(8)	-C(67)	1.479(10)	C(62)	-C(63)	1.404(10)
N(8)	-Li(2)	2.060(13)	C(63)	-C(64)	1.391(13)
N(9)	-C(68)	1.476(12)	C(64)	-C(65)	1.362(14)
N(9)	-C(69)	1.451(12)	C(65)	-C(66)	1.373(12)
N(9)	-C(70)	1.462(12)	C(67)	-C(68)	1.447(12)
N(9)	-Li(2)	2.046(13)	C(74)	-C(75)	1.505(10)
N(10)	-C(74)	1.335(10)	C(75)	-C(76)	1.383(11)
N(11)	-C(74)	1.344(9)	C(75)	-C(80)	1.379(11)
N(11)	-C(81)	1.479(10)	C(76)	-C(77)	1.358(12)
N(11)	-Li(2)	2.044(13)	C(77)	-C(78)	1.399(13)
N(12)	-C(82)	1.499(12)	C(78)	-C(79)	1.361(13)
N(12)	-C(83)	1.456(12)	C(79)	-C(80)	1.387(12)
N(12)	-C(84)	1.433(12)	C(81)	-C(82)	1.456(12)

Bond angles (deg.)

N(7)	-Y(2)	-N(8)	55.7(2)	C(68)	-N(9)	-C(69)	111.0(7)
N(7)	-Y(2)	-N(10)	145.0(2)	C(68)	-N(9)	-C(70)	109.0(7)
N(7)	-Y(2)	-N(11)	99.86(18)	C(68)	-N(9)	-Li(2)	101.3(6)
N(7)	-Y(2)	-C(43)	108.4(3)	C(69)	-N(9)	-C(70)	109.6(7)
N(7)	-Y(2)	-C(50)	92.4(2)	C(69)	-N(9)	-Li(2)	108.3(6)
N(7)	-Y(2)	-C(60)	28.9(2)	C(70)	-N(9)	-Li(2)	117.4(6)
N(7)	-Y(2)	-C(74)	128.0(2)	Y(2)	-N(10)	-Si(4)	135.2(3)
N(8)	-Y(2)	-N(10)	93.8(2)	Y(2)	-N(10)	-C(74)	92.4(4)
N(8)	-Y(2)	-N(11)	83.2(2)	Si(4)	-N(10)	-C(74)	127.7(6)
N(8)	-Y(2)	-C(43)	100.1(2)	Y(2)	-N(11)	-C(74)	90.0(4)
N(8)	-Y(2)	-C(50)	147.0(2)	Y(2)	-N(11)	-C(81)	132.6(4)
N(8)	-Y(2)	-C(60)	29.0(2)	Y(2)	-N(11)	-Li(2)	86.1(4)
N(8)	-Y(2)	-C(74)	95.8(2)	C(74)	-N(11)	-C(81)	120.5(5)
N(10)	-Y(2)	-N(11)	55.4(2)	C(74)	-N(11)	-Li(2)	119.3(6)
N(10)	-Y(2)	-C(43)	92.4(3)	C(81)	-N(11)	-Li(2)	105.1(6)
N(10)	-Y(2)	-C(50)	112.9(3)	C(82)	-N(12)	-C(83)	107.3(7)
N(10)	-Y(2)	-C(60)	122.8(2)	C(82)	-N(12)	-C(84)	112.6(7)
N(10)	-Y(2)	-C(74)	28.6(2)	C(82)	-N(12)	-Li(2)	99.8(6)
N(11)	-Y(2)	-C(43)	147.8(3)	C(83)	-N(12)	-C(84)	108.8(7)
N(11)	-Y(2)	-C(50)	95.9(2)	C(83)	-N(12)	-Li(2)	114.2(6)
N(11)	-Y(2)	-C(60)	99.5(2)	C(84)	-N(12)	-Li(2)	113.8(6)
N(11)	-Y(2)	-C(74)	28.8(2)	Y(2)	-C(43)	-C(44)	122.8(5)
C(43)	-Y(2)	-C(50)	97.9(3)	C(43)	-C(44)	-C(45)	123.3(7)
C(43)	-Y(2)	-C(60)	98.1(3)	C(43)	-C(44)	-C(49)	122.5(7)

C(50)	-Y(2)	-C(74)	98.9(3)	C(45)	-C(46)	-C(47)	121.8(8)
C(60)	-Y(2)	-C(74)	120.4(2)	C(46)	-C(47)	-C(48)	117.7(7)
N(7)	-Si(3)	-C(57)	111.2(3)	C(47)	-C(48)	-C(49)	120.7(8)
N(7)	-Si(3)	-C(58)	107.5(3)	C(44)	-C(49)	-C(48)	123.0(7)
N(7)	-Si(3)	-C(59)	113.5(3)	Y(2)	-C(50)	-C(51)	115.5(6)
C(57)	-Si(3)	-C(58)	107.7(4)	C(50)	-C(51)	-C(52)	123.9(8)
C(57)	-Si(3)	-C(59)	109.3(3)	C(50)	-C(51)	-C(56)	122.5(8)
C(58)	-Si(3)	-C(59)	107.4(4)	C(52)	-C(51)	-C(56)	113.4(8)
N(10)	-Si(4)	-C(71)	115.5(4)	C(51)	-C(52)	-C(53)	122.0(9)
N(10)	-Si(4)	-C(72)	108.8(3)	C(52)	-C(53)	-C(54)	120.9(9)
N(10)	-Si(4)	-C(73)	107.6(4)	C(53)	-C(54)	-C(55)	117.7(10)
C(71)	-Si(4)	-C(72)	110.6(4)	C(54)	-C(55)	-C(56)	123.(1)
C(71)	-Si(4)	-C(73)	106.0(4)	C(51)	-C(56)	-C(55)	122.9(9)
C(72)	-Si(4)	-C(73)	108.0(4)	Y(2)	-C(60)	-N(7)	61.0(4)
Y(2)	-N(7)	-Si(3)	137.2(3)	Y(2)	-C(60)	-N(8)	62.5(4)
Y(2)	-N(7)	-C(60)	90.1(4)	Y(2)	-C(60)	-C(61)	151.6(5)
Si(3)	-N(7)	-C(60)	129.7(5)	N(7)	-C(60)	-N(8)	116.5(6)
Y(2)	-N(8)	-C(60)	88.5(4)	N(7)	-C(60)	-C(61)	122.3(6)
Y(2)	-N(8)	-C(67)	131.9(5)	N(8)	-C(60)	-C(61)	121.2(7)
Y(2)	-N(8)	-Li(2)	85.6(4)	C(60)	-C(61)	-C(62)	119.7(6)
C(60)	-N(8)	-C(67)	120.4(6)	C(60)	-C(61)	-C(66)	121.0(6)
C(60)	-N(8)	-Li(2)	119.9(6)	C(62)	-C(61)	-C(66)	119.3(6)
C(67)	-N(8)	-Li(2)	106.8(6)	C(61)	-C(62)	-C(63)	121.0(7)
C(62)	-C(63)	-C(64)	118.0(7)	C(76)	-C(75)	-C(80)	119.9(7)
C(63)	-C(64)	-C(65)	121.3(8)	C(75)	-C(76)	-C(77)	120.1(7)
C(64)	-C(65)	-C(66)	120.3(9)	C(76)	-C(77)	-C(78)	120.6(8)
C(61)	-C(66)	-C(65)	119.9(7)	C(77)	-C(78)	-C(79)	119.0(7)
N(8)	-C(67)	-C(68)	110.2(6)	C(78)	-C(79)	-C(80)	121.0(8)
N(9)	-C(68)	-C(67)	115.3(8)	C(75)	-C(80)	-C(79)	119.4(7)
Y(2)	-C(74)	-N(10)	59.0(4)	N(11)	-C(81)	-C(82)	108.8(6)
Y(2)	-C(74)	-N(11)	61.2(3)	N(12)	-C(82)	-C(81)	113.6(8)
Y(2)	-C(74)	-C(75)	153.6(5)	N(8)	-Li(2)	-N(9)	87.2(5)
N(10)	-C(74)	-N(11)	114.4(6)	N(8)	-Li(2)	-N(11)	104.7(6)
N(10)	-C(74)	-C(75)	124.9(7)	N(8)	-Li(2)	-N(12)	129.6(6)
N(11)	-C(74)	-C(75)	120.6(7)	N(9)	-Li(2)	-N(11)	125.5(6)
C(74)	-C(75)	-C(76)	120.5(7)	N(9)	-Li(2)	-N(12)	123.8(6)
C(74)	-C(75)	-C(80)	119.6(6)	N(11)	-Li(2)	-N(12)	88.9(5)

Part IV: Structure determination of $[PhC(NSiMe_3)(CH_2)_3NMe_2]_2YCH_2Ph.(pentane)_{0.5}$ (4).

Abstract. $C_{37}H_{59}N_6Si_2Y.(C_5H_{12})_{0.5}$, M = 769.07, triclinic, P , a = 8.762(3), b = 11.350(2), c = 22.234(4) Å, α = 80.56(1)°, β = 89.39(2)°, γ = 79.65(2)°, V = 2145.3(9) Å^3, Z = 2, D_x = 1.196 g cm^{-3}, λ(MoK) = 0.71073 Å, μ = 14.5 cm^{-1}, $F(000)$ = 816, T = 130 K, $wR(F^2)$ = 0.1761 for 7554 reflections with $F_o^2 \geq 0$ and 449 parameters and $R(F)$ = 0.0586 for 5784 reflections obeying $F_o \geq 4.0\ \sigma(F_o)$ criterion of observability.

The asymmetric unit consists of one molecule of the title compound and a half (disordered) pentane solvent molecule (on an inversion center).

Experimental

X-ray diffraction: Crystal and Molecular Structure.

The crystal, a parallelepiped of approximate size 0.20 x 0.25 x 0.50 mm., used for characterization and data collection was glued on top of a glass fiber by using inert-atmosphere handling techniques and was transferred into the cold nitrogen cold stream of the low temperature unit[1] mounted on an Enraf-Nonius *CAD-4F*[2] diffractometer, interfaced to a *INDY* (*Silicon Graphics*) *UNIX* computer (Mo tube, 50 kV, 40 mA, monochromated Mo-K radiation, $\Delta\omega = 0.90 + 0.34$ tg θ).

Unit cell parameters[3] and orientation matrix were determined from a least-squares treatment of the *SET4*[4] setting angles of 22 reflections in the range $16.52 < \theta < 20.42°$. The unit cell was identified as triclinic, space group P . Reduced cell calculations did not indicate any higher metric lattice symmetry[5] and examination of the final atomic coordinates of the structure did not yield extra metric symmetry elements.[6,7]

The intensities of three standard reflections, monitored every three hours of X-ray exposure time, showed no greater fluctuations during data collection than those expected from Poisson statistics. A 360° ψ-scan for a reflection close to axial (212) showed a variation in intensity of less than 12% about the mean value. Intensity data were corrected for Lorentz and polarization effects, scale variation, but not for absorption and reduced to F_o^2.[8]

The structure was solved by Patterson methods and extension of the model was accomplished by direct methods applied to difference structure factors using the program *DIRDIF*.[9] The positional and anisotropic thermal displacement parameters for the non-hydrogen atoms were refined. Refinement was complicated by a disorder problem: from the solution it was clear that the pentane solvent molecule was highly disordered over a inversion center; the center atom was located at the inversion center. The electron density of the pentane carbon atoms appeared to be spread out, indicating conformational disorder. A disorder model for C38 (50:50) was used in the refinement.

riding on their carrier atoms with their

of their parent atom, where c = 1.2 for the aromatic / non-methyl hydrogen atoms and c = 1.5 for the methyl hydrogen atoms and where values U_{equiv} are related to the atoms to which the H atoms are bonded. The methyl-groups were refined as rigid groups, which were allowed to rotate free.

Final refinement on F^2 carried out by full-matrix least-squares techniques converged at $wR(F^2)$ = 0.1761 for 7554 reflections with $F_o^2 \geq 0$ and $R(F)$ = 0.0586 for 5784 reflections with $F_o \geq 4.0\ \sigma(F_o)$ and 449 parameters.

The final difference Fourier map was essentially featureless with a few peaks of max. 1.025(11) e/Å^3 within 1.0 Å from Y. No other significant peaks having chemical meaning above the general background were observed in the final difference Fourier syntheses.

The positional and anisotropic thermal displacement parameters for the non-hydrogen atoms and isotropic thermal displacement parameters for hydrogen atoms were refined on F^2 with full-matrix least-squares procedures minimizing the function $Q = \Sigma_h\ [w(|F_o^2 - kF_c^2|)^2]$, where $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$, $P = [\max(F_o^2,0) + 2F_c^2] / 3$, F_0 and F_c are the observed and calculated structure factor amplitudes, respectively; a and b were refined. Reflections were stated observed if satisfying $F^2 > 0$ criterion of observability.

Neutral atom scattering factors and anomalous dispersion corrections were taken from *International Tables of Crystallography.*[10] All calculations performed on the HP9000/735 computer at the University of Groningen with the program packages *SHELXL*[11] (least-square refinements), *PLATON*[12] (calculation of geometric data and the *ORTEP* illustrations) and a locally modified version of the program *PLUTO*[13] (preparation of illustrations).

Each asymmetric unit contains one formula unit molecule with no atom setting at special position and an half disordered pentane solvent molecule with the center C-atom located at sites with symmetry. The triclinic unit cell contains two discrete units of the title compound and one highly disordered pentane molecule separated by normal van der Waals distances.[16]

No missed symmetry (*MISSYM*) or solvent-accessible voids were detected by procedures implemented in *PLATON.*[17,18]

The structure contains no potential residual solvent accessible voids.[19]